1995

Formation of austenite grain structure accompanying hot rolling of Nb-V and Nb-V-Ti microalloyed steels

Muhammad Irfan

University of Wollongong

FORMATION OF AUSTENITE GRAIN STRUCTURE ACCOMPANYING HOT ROLLING OF Nb-V AND Nb-V-Ti MICROALLOYED STEELS

A Thesis Submitted In Fulfilment Of
The Requirement For The Degree Of
Honours Master Of Engineering

by

MUHAMMAD IRFAN, Ir. (ITB)

DEPARTMENT OF MATERIALS ENGINEERING
THE UNIVERSITY OF WOLLONGONG
AUSTRALIA

1995

ACKNOWLEDGEMENTS

I would like to express my most sincere thanks and appreciation to my thesis supervisor, Professor Tara Chandra for his skillful guidance, great enthusiasm, encouragement and valuable comments throughout the course of this investigation.

I am specially grateful to Ir. Fazwar Bujang, MM, Ir. Djoko Subagyo and Ir. Soelaiman MS, MM, for provision of the experimental materials, technical information and financial support.

I wish to thank Professor. D.P. Dunne for providing laboratory facilities and his genereous encouragement. I wish to thank R. Kinnell, G. Tillman and Ir. Tri Djaka, MEng. for their help in my experiments on mini hot rolling in The Department of Material Engineering laboratory in The University of Wollongong, Australia.

Also , I wish to thank Professor Nick Standish for his skillful guidance and comments on my english.

Finally, it is my great pleasure to thank my wife Ima and my son Reza for their patience, understanding and constant encouragement.

MUHAMMAD IRFAN

Cilegon, 1995

ABSTRACT

An investigation of the grain coarsening characteristics has been carried out on samples of commercial C-Mn, Nb-V and Nb-V-Ti steels produced at PT. Krakatau Steel, Indonesia. The results have shown that the grain coarsening occurs at a low temperature in the cast C-Mn steel, which seems to be due to the low temperature of solution of the AlN grain boundary pinning particles. It was found that the grain coarsening temperature (GCT) can be raised by microalloy additions of Nb-V and Nb-V-Ti. Austenite structural evolution after single-pass rolling has also been investigated in the above steels, in the range of reductions from 20% to 60% and at temperatures of 950°C - 1150°C. The experimental procedure involved first heating the samples to 1200°C for 15 minutes, cooled to various temperatures (950°C - 1150°C), rolled and then subsequently quenching after the samples were held at the selected temperature for different times (3 - 1800 sec.).

The experimental results show that the rate of recrystallization in the C-Mn, Nb-V and Nb-V-Ti steels increased with increase in rolling temperature or reduction. The addition of micro alloying increased the critical rolling reduction, and critical rolling reduction decreased with an increase in rolling reduction and holding temperature. For a given holding time, the mean recrystallized austenite grain size, increased with increasing temperature of deformation and decreased with increased amount of reduction.The Nb-V and Nb-V-Ti steels studied showed only a slight increase in recrystallized grain size on holding which seems to be due to pinning effect of undissolved alloy carbonitride particles.

CONTENT

CHAPTER 1

INTRODUCTION

INTRODUCTION

Thermomechanical processing is a technique designed to improve the mechanical properties of materials by controlling the hot-deformation processes (1). The purpose of thermo-mechanical processing is to control the structure, morphology and grain size of the austenite in HSLA steels so that the eventual transformation of the austenite produces the optimum ferritic structure. The reheating temperature controls the initial austenite grain size by controlling the solution and precipitation of the microalloy carbides and nitrides. The initial austenite grain structure prior to hot rolling should be uniformly fine for ease of hot rolling and to promote a unifomly ferrite fine grain structure. Refinement of which consists mainly of two steps : (a). the deformation of austenite above the recrystallization temperature and/or (b). deformation of the austenite below the recrystallization temperature by hot rolling. An effective way to obtain a fine and uniform ferrite grain structure is to attain recrystallized austenite grains as fine as possible, followed by a large amount of deformation in the non- recrystallization region. Controlled rolling, controlled-cooling and direct-quenching are typical examples of thermomechanical processing. Such processing saves energy in steel manufacture by minimizing or even eliminating the heat treatment after hot-deformation, thus increasing productivity for high grade steels, (1).

The purpose of controlled rolling is to obtain a uniform, fine-grained structure in the hot-rolled condition and thereby to produce steel with high strength, good toughness at low temperature, and superior weldability. In order to attain these properties in hot rolled steel, the chemistry, slab-reheating temperature,hot-rolling, transformation behaviour, and cooling rate must be properly controlled. Conventional controlled rolling involves the control of hot-rolling conditions alone. The modern controlled rolling process, however, covers, the whole process from slab-reheating and hot-rolling to controlled cooling. Modern controlled rolling can produce not only a fine-grained ferrite structure, but acimular ferrite and dual-phase structure, (2,3).

The purpose of the present work was to investigate the austenite formation from deformated austenite in Nb-V and Nb-V-Ti steel samples and C-Mn steel for comparison with rolling variables such as rolling temperature, amount of reduction and holding time. The initial grain size can be investigated with grain coarsening temperature as reference before rolling condition. Further purpose of this work was to examine the effect of rolling temperature, effect of amount of reduction and holding time on the austenite formation.

CHAPTER 2

REVIEW OF LITERATURE

2. REVIEW OF LITERATURE

2.1. EFFECT OF MICROALLOYING ELEMENTS

2.1.1. General Effect Of Microalloying Element

The most importan microalloying element in HSLA steel are niobium, vanadium and titanium. The main effect of microalloying elements in HSLA steel is to achieve improved strength, toughness and ductility for low carbon steel. The key to this development rests on the refinement of the ferrite grain size produced by the austenite to ferrite transformation and the precipitation hardening of carbides, nitrides, or carbonitrides of these elements (4,5,6).

These elements (Nb. V, Ti) have different affinities for carbon and nitrogen. (Figure 2.1).The solubility of their carbonitrides in austenite and the capability to precipitates fine-dispersed particles in austenite and ferrite provide a basis for strengthening. (1,7,8,9,10).

At the reheating temperature, the undissolved carbide and nitrides precipitates contribute to refinement of the initial austenite grains. In the reheating temperature range 1100 - 1250°C, titanium nitride is more stable than niobium nitride and vanadium nitride, while vanadium nitride is the most soluble in austenite, (5,11,10).Titanium nitride is more stable than titanium carbide, and they have diferent solubility in austenite. The very fine and dispersed titanium nitride particles can substantially increase the austenite grain coarsening temperature and exert a strong effect on ferrite grain refinement.

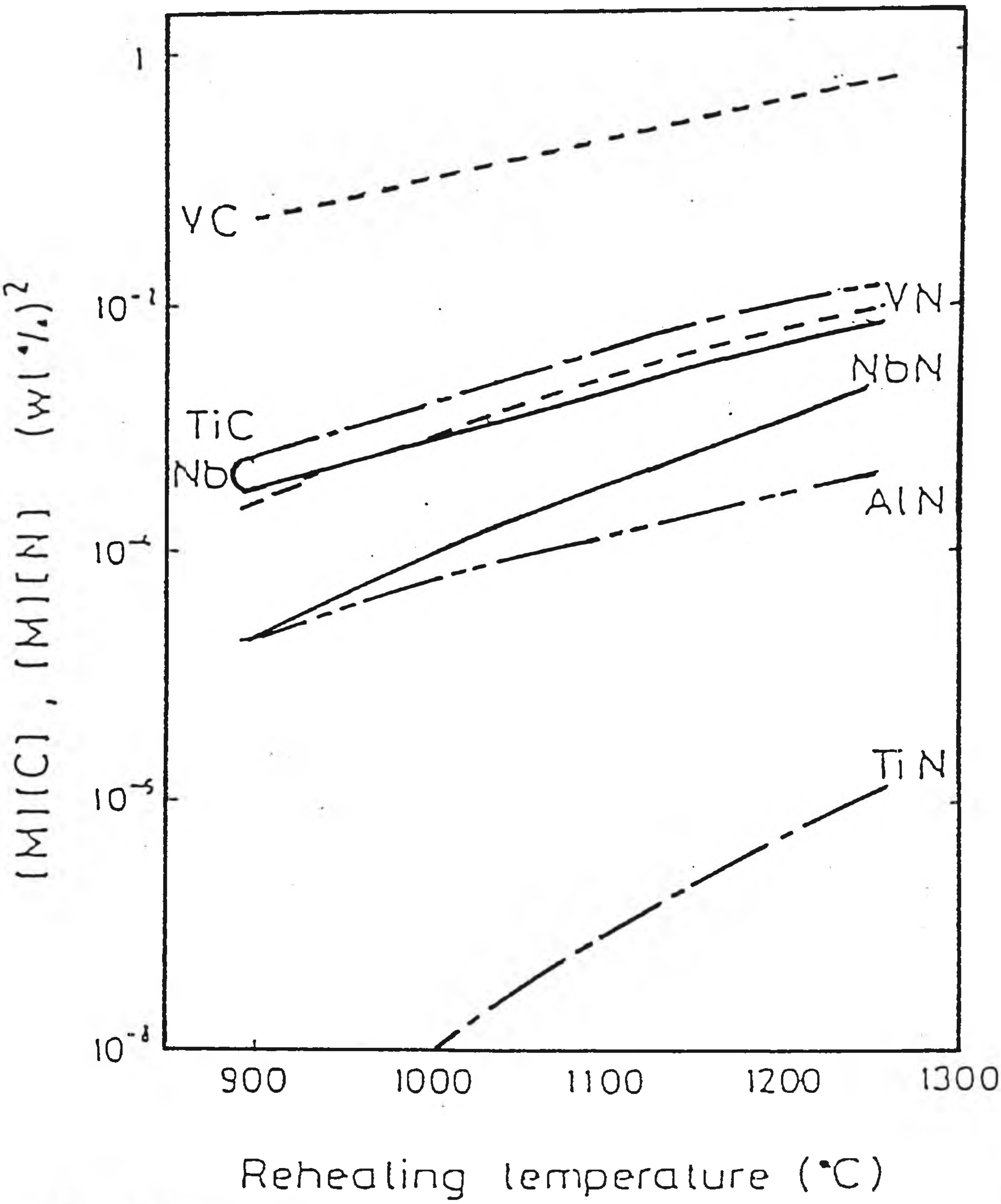

Figure 2.1. **Solubility Products of Carbides and Nitrides in Austenite.**

Therefore, this perticles can control the austenite grain size of reheated slab. Small concentration of titanium is consumed mainly in titanium nitride formation. Titanium nitride is virtually insoluble in austenite, as a result it plays little part in precipitation strengthening. With increasing amounts of titanium, strengthening of hot rolled products increase due to its strong precipitation strengthening rather than grain refinement strengthening. Titanium reacts with intertitial elements such as, nitrogen, carbon and including oxygen and sulfur to from a number of compounds in low carbon steel. Some of these compounds have desirable effects, but the precence of others are undersirable (1,5,10,13).

2.1.2. Effect Of Titanium

In low carbon steel, titanium reacts with interstitial element to form compounds such as nitride. Titanium nitride is stronger than titanium carbide, and they also have different solubility in austenite. Titanium nitride is known to be effective in inhibiting austenite grain growth at high temperature (10).

The very fine and dispered titanium nitride particles can increase the austenite grain coarsening temperature and refine the austenite grain size during thermomechanical processing of titanium-bearing steels. This effect is generally ascribed to the presence of titanium-rich precipitation (10,14,15). Small concentration of titanium are consumed mainly in titanium nitride formation. The titanium nitride is virtually insoluble in austenite, produce fine grain where palys little part in precipitation strengthening (1,13).

2.1.3. Effect Of Niobium

Niobium can interacts with carbon or nitrogen to form small particles as carbide, nitride or carbonitride. Not only the formation of small particles depend on solubility carbide nitride in austenite, but also the retardation of austenite recrystallization, grain growth during normalizing process of controlled rolling process. Those small perticles will couse grain refinement of austenite and finally ferrite, (10,15,16,17).

In niobium steels, the onset of austenite recrystallization is greatly retarded by decrease in temperature. Although solute niobium atoms and lower temperature retard the onset of austenite recrystallization, they do not retard the progress of austenite recrystallization. When strain induced precipitation occurs, the onset of austenite recrystallization is retarded markedly, and strain induced precipitation can retard both the onset and progress austenite recrystallization, (14,18).

Niobium carbide and nitride dissolved in austenite at reheating temperature, but subsequently reprecipitate during controlled rolling at lower temperature. The solubility of the niobium nitride and niobium carbide in austenite is different. Niobium is mostly consumed in niobium nitride formation (1,4,16.19). The fine particles of niobium nitride and carbide precipitated during or after transformation in austenite or ferrite will cause grain refinement and precipitation hardening resulting the increase of strength.

2.1.4. Effect Of Vanadium

Vanadium, as niobium and titanium, also can interact with carbon and nitrogen to form carbide and nitride, but this interaction is not stronger as than of niobium carbides and nitrides. Vanadium also controls austenite recrystallization and increase the strength caused by precipitation hardening, especially for steels with high nitrogen content. This element can produce similar effects with niobiumn except that the solution temperature is lower and recrystallization retardation is weaker than that of the niobium, (1,4,15,20,21).

Erasmus (22) studied the effect of vanadium on the grain coarsening characteristic of austenite, and found a progressive increase in grain coarsening temperature with increasing vanadium content. Small additions of vanadium are relatively more effective than large additions. Also grain coarsening always started at temperature well below the solubility limit of vanadium nitride. The austenite grain boundary migration will be inhibited by niobium carbonitrides and vanadium carbonitrides during reheating. (5,22,23).

Vanadium is a very strong nitrogen fixer, therefore major part of the strengthening in vanadium bearing steel is result of the precence of vanadium nitride precipitates. Although vanadium has a grain refining effect, but this vanadium grain refinement does not make a very large contribution to the strengthening of vanadium steels. (10,15,20,21).

2.1.5. Solubility Carbides And Nitrides In Austenite

Niobium, vanadium and titanium have different affinities with carbon and nitrogen in austenite, and cause the different solubility products of carbide and nitride in respective microalloying elements. In Figure 2.1, solubility products of carbide and nitride in austenite are mostly at temperature range for slab reheating (1100 - 1250°C), titanium nitride and niobium nitride are the most stable compound ands vanadium carbide the most soluble into austenite(1,24).

While niobium carbide and titanium carbide lie between the two compounds. Niobium and titanium content disolved in austenite at reheating temperature can be varied widely, depending on the temperature and carbon content. All nitrides have lower solubilities in austenite than the respective carbides (24).

In titanium-bearing steels, titanium nitride is first formed and after all the nitrogen is combined as titanium nitride, titanium carbide may subsequently precipitate with increse titanium content. (55).

The interaction vanadium with carbon and nitrogen will be form vanadium carbon and nitride but is not as stable as niobium nitride and carbide. This means that vanadium precipitates austenite at a lower temperature than that titanium and niobium (1,10,15,17.21).

2.1.6. Retardation Of Austenite Recrystallization

The retardation of austenite recrystallization by microalloy additions in HSLA steels is considered to be due to two mechanisms. The first is the grain boundary pinning after of strain-induced precipitates and the second is the solute drag coused by the microalloying element in solution.

The retardation of recrystallization by microalloying additions can be attributed to their presence as solute or precipitates or to a combination of the two. The lower temperature and smaller amounts of deformation can contribute to retardation of recrystallization, (25).

The retarding effect of dissolved solute atoms on recrystallization has been found to increase with increasing strain introduced by the solute into the austenite lattice influencing the dislocation solute atom interaction, (14,26,27).

When a dislocation or a boundary moves, the solutes can increase substantial a dragging force opposing recrystallization. Solute can also lower the stacking foult energy, hinder recovery and consequently recrystallization is delayed or can be prevented completely (27,28).

In addition, the retardation of recrystallization in HSLA steels depends on the intensity of the precipitation which in turn depends on the applied strain and on the steel composition. The intensity of precipitation also depends on the solubility of the microalloy carbonitrides (18).

Further, the intensity of precipitation depends on the stoichiometric ratio of the microalloying addition to carbon or nitrogen present in the steel and the intensity of the strain-induced precipitation will be highest if the composition is near to stoichiometric ratio, (18,19).

The solute niobium atoms retard recovery and recrystallization until the occurance of strain induced precipitation, while strain induced precipitates can retard both the onset and progress of austenite recrystallization. (6,15,19).

In vanadium steels the retardation effect by solute atoms is weak, recrystallization is complete before the occurance of strain induced precipitation, and a significant retarding effect on austenite recrystallization can not be expected. Although vanadium is a strong carbide-forming element, it has a slight recovery-retarding effect, whereas titanium exerts a very strong recrystallization-retarding effect on deformed austenite (14,15,18).

A small concentration of titanium is consumed in titanium nitride formation. The titanium nitride is commonly present as large cube shaped particle that is formed in the melt and this titanium makes no contribution towards strength, because the nitride is virtually insoluble in austenite, it plays no part in precipitation strengthening. Hence, a higher titanium content than the stoichiometric ratio is needed if precipitation strengthening by titanium carbide is required.

2.2. THE STAGES OF CONTROLLED ROLLING PROCESS

The modern controlled rolling is comprised of three stages of processing as proposed by Tanaka (29), and given in Figure 2.2.

(I)Deformation in the austenite-recrystallization region.

(II) Deformation in the non-recrystallization region.

(III) Deformation in the austenite-ferrite two-phase region

Stage1 : Coarse austenite, (a), is refined by repeated deformation and recrystallization, (b), but it still transforms to relatively coarse ferrite mixed with coarse bainite, (b’).

Stage 2 : Deformation bands are formed in elongated unrecrystallized austenite grains, (c'), and ferrite nucleateson the deformation bands as well as austenite grain boundary, leading to fine ferrite grains, (c').

Stage 3 : Deformation in the austenite-ferrite two-phase region cintinues from stage 2, and also deforms the ferrite producing subgrains structure (d'). Microstructure, (b'), and (c'), consist of equiaxed grain alone, whereas, (d'), consist of equiaxed soft grains and elongated hard grains with subsructure within them.

Accelereted cooling following hot-rolling has a strong effect on microstructure. When finish rolled in the high temperature austenite region and subsequently air-cooled. The Microstructure is a mixture consisting of coarse ferrite and fine ferrite grains dispersed with bainite/martensite islands (14,30).

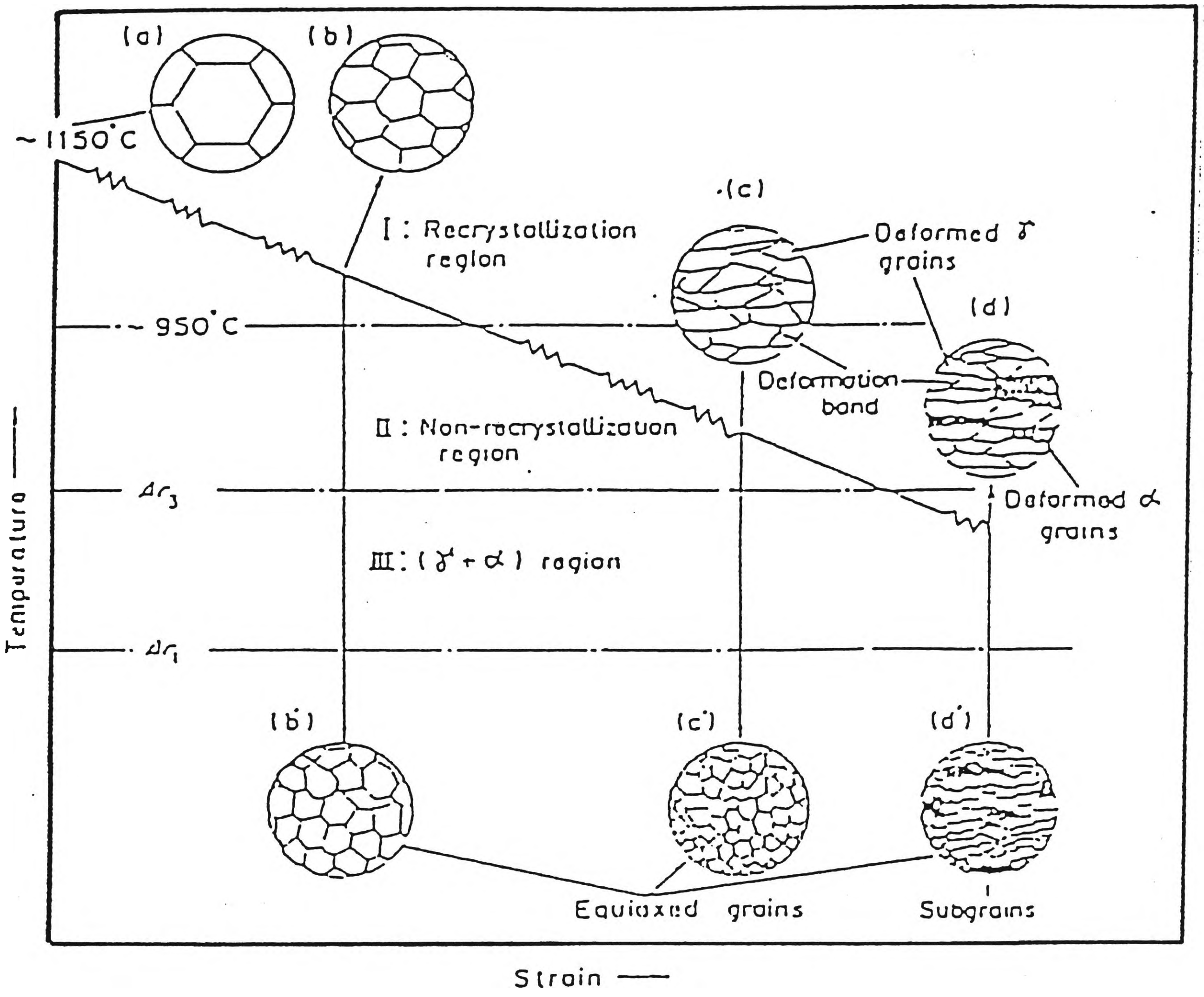

Figure 2.2. Schematic Illustration of Three Stages of Controlled Rolling Process and Change in Microstructure with Deformation in each Stage.

2.3. RECRYSTALLIZATION MECHANISM

2.3.1. Recrystallization

Two nucleation mechanisms have been identified for recrystallization. The first is strained-induced boundary migration, where strained-free nuclei moves into the neighbouring region leaving a strain-free recrystallization region behind. The boundary moves into the grain which contains the higher dislocation density in local region.

In the second nucleation mechanism new grain boundaries are formed in regions of sharp lattice curvature through sub-grain growth. This mechanism seems to predominate at high strains, with nuclei appearing at grain boundaries, twin boundaries, or at inclusion or second phase particles. The nuclei form only in region of in-homogeneous deformation, (4,7).

The recrystallization of austenite may be dynamic or static. It is reported (8) that when dynamic recrystallization occurs the austenite grain size is not effected by the initial grain size or amount of deformation. In the static recrystallization, the recrystallized grain size is assumed to be determined by the number of nucleation sites, which depend on the interfacial area of austenite per unit volume, austenite grains become elongated with increased rolling reduction and deformation bands are introduced within the grains (8,9).

2.3.2. Static Recrystallization of Austenite

The driving force for static recrystallization is the stored energy of deformation, but a critical or minimum deformation is required to produce static recrystallization. A high temperature of deformation enhances the rate of recrystallization. (8,20,22,31).

The rate of recrystallization and the resulting grain size are sensitive, both to rolling variable, particularly pass reduction and temperature, and to other metallurgical variables. In microalloyed steel the metallurgical variables are prior austenite grain size, microalloy content in solution and the state of precipitation of nitrides and carbonitrides in austenite (5,10,32,33).

The statically recrystallized austenite grain size is independent of deformation temperature and is refined by decrease in the starting grain size, (9,22,32,33).

2.3.3. Dynamic Recrystallization.

The condition favouring dynamic recrystallization are deformation at high temperature, large reductions, and low strain rate. The size of the dynamically recrystallized grains is determined both by the temperature and strain rate but does not depend on the initial grain size. (22,33,34,35).

At the high reduction and high deformation temperature condition, grain size is determined primarily by the rolling temperature. At intermediate reductions and temperatures, it is determined mainly by the amount of reduction during rolling.

Grain refinement at high temperature is atribute to dynamic recrystallization (8,20). The onset of dynamic recrystallization is marked by a decrease in flow stress during deformation. The critical strain for dynamic recrystallization is somewhat lower than the peak strain, (ϵ_p), the strain at the maximum stress referred to as the peak strain (1,7).

2.4. EFFECT OF DEFORMATION ON AUSTENITE RECRYSTALLIZATION

2.4.1. Deformation in Austenite Recrystallization Region

When rolling is applied at a temperature higher than a critical value in the stable austenite region (>A_r3), austenite grains can be refined by repeated deformation and recrystallization. And when deformation at a temperature below the critical value, recrystallization of deformed austenite will not occur, deformed austenite grains retain the as-deformed alongated shape, and deformation band form in unrecrystallized austenite grains, (9,30,33).

After deformation at temperature higher than the critical value in the austenite recrystallization region, static recrystallization start when the strain exceeds that corresponding to the critical reduction which is determined by the prior deformation conditions and the original grain size (36). Recrystallization rate increase with the increase of strain and temperature of deformation (37). The size of statically recrystallized austenite grains is almost independent of the deformation temperature, and is refined by decrease of initial grain size and increase of reduction (29).

The purpose of deformation in the austenite recrystallization region is to reduce the austenite grain size as much as possible by repeated deformation and recrystallization. If the rolling reduction per pass is smaller than the critical reduction for recrystallization, but are high enough to cause partial recrystallization during the time intervals of rolling passes the volume fraction of austenite recrystallization will increase with number of rolling pass. Complete and uniform recrystallized austenite structure can be obtained by multi-pass rolling. (1,30,38).

2.4.2. Deformation On Of Austenite In The Non-Recrystallization Region

The purpose of deformation in the non- recrystallization region is to increase ferrite-nucleation site by producing deformation bands in the interiors of elongated austenite grains. The fundamental difference between conventionally hot-rolledsteel and controlled rolled steel is that in the former ferrite grains nuclease excessively at austenite grain boundaries, but in controlled rolling ferrite grains nucleate at austenite grain boundaries as well as grain interior.

Deformation bands in deformed austenite increase the opportunity for ferrite nucleation, and are therefore similar in this respect to the austenite grain

boundaries, and producing a grain structure that form recrystllized austenite grains (1,30). The essence of controlled rolling is to deform austenite in the non-recrystallization region that deformation bands are introduced within the austenite grains dividing one austenite grain into several blocks, thus increasing
the density of nucleation sites for ferrite grains and producing refinement of the ferrite grain. (1,3).

The variation in austenite grain structure accompanying rolling in the non recrystallization region, especially with decrease in temperature, makes recrystallization become more difficult and it is vertually suppressed below 950°C. With increasing amount of deformation in this non recrystallization region austentite grains become elongated and deformation band are generated within them (39).

2.4.3. Deformation In Austenite-FerriteTwo Phase Region

The limited extent of grain refinement due to deformation in the austenite recrystallization and non- recrystallization regions can be overcome by rolling in the two-phase region. The steel deformation in the two phase region produces a mixed grain structure, which consist of equiaxed ferrite grains and cold worked grains in term of an optical microscopic scale. Deformation in the austenite region alone produces microstructure consisting of equiaxed ferrite grain with low dislocation density (1,30). Deformation in the austenite-ferrite two phase region has the purpose of increase strength, in particular tensile strength. Tensile strength increases linearly with volume fraction of deformed ferrite, but the increase in plain C-Mn steel is smaller than in micro alloying bearing steel (1,3).

CHAPTER 3

EXPERIMENTAL PROCEDURE

3. MATERIALS AND EXPERIMENTAL PROCEDURE

3.1. MATERIALS

The Nb-V and Nb-V-Ti steels were supplied by PT. KRAKATAU STEEL, in the form of continuous cast slabs. The chemical compositions are given in Table 3.1. It should be noted that the inclusion of the C-Mn steel was as a reference material

Table 3.1. Chemical Compositions of Steels Investigated (wt %)

ELEMENT	C	Si	Mn	P	S	AL	N	Nb	V	Ti
C-Mn	0.084	-	0.326	0.003	0.008	0.014	0.006	-	-	-
Nb-V	0.096	0.240	1.130	0.007	0.006	0.031	0.007	0.030	0.050	-
Nb-V-Ti	0.096	0.364	1.480	0.009	0.006	0.063	0.007	0.050	0.030	0.020

3.2. EXPERIMENTAL PROCEDURE

3.2.1. Grain Coarsening Experiments

Samples were taken from the quarter width and quarter location of slabs to avoid the impurity segregation zone in the slab center thickness location and surface inperfaction. The samples were 15 mm high x 15 mm wide and 30 mm in thickness, rectangular of shape. The samples were isochronally annealed for 15 minutes at various austenizing temperatures, ranging from 950°C to 1250°C, in argon atmosphere to prevent excessive oxidation. The samples were quenched in water immediatly after annealing.

3.2.2. Metallography.

The samples were first mechanically polished, and then etched to delinetate prior austenite grain boundaries in martensite. A saturated aqueous picric acid solution was used for etching. The etchant had the following composition : 100 ml water with 5% saturated picric acid plus 20 drops of wetting agent (Teepol) and 20 drops of concentrated HCL. The etchant solution was heated to 60°C - 70°C and the etching time was varied (30 - 60 sec.) depending on the composition and the austenization temperature.

3.2.3. Grain Size Measurement

The intercept circle method was employed in determining the average austenite grain size using a micrometer eyepiece attached to an optical microscope. The number of grain boundaries intercepting the test circle was counted by using the amount of circumference of test circle devided by number of grain boundaries intercepted by the circle line. A minimum of 5 fields per specimen were observed, fields were taken along the center line of the samples. 95% confidence limits were calculated for all grain size by using the ASTM E 112-88 procedure "Standard Test Method For Determining Average Grain Size",. The measured values were plotted to correlate austenite grain size with austenizing temperature, so that the grain coarsening temperature could be evaluated.

3.2.4. Hot Rolling

The C-Mn, Nb-V and Nb-V-Ti steels were used for single- pass rolling and the rolling experiments were done in the Material Engineering Department, the University of Wollongong, Australia. For all three types of steels, stepped rolling specimens, with the dimensions given in Figure 3.1, were machined from quarter width and thickness position which allowed simultaneous reduction of 20%, 40% and 60% under single rolling pass.

The samples were reheated for 15 minutes in an argon atmosphere at different austenitizing temperatures of : 950°C, 1000°C, 1050°C, 1100°C, and 1150°C, and rolled in two-high rolling mill. The rolled samples were either water quenchend directly after rolling (within 3 sec.), or transferred to a furnace set at the predetermined temperature. The samples were held at this temperature for 30, 300 and 1800 sec. before quenching. The rolling schedule is shown diagramatically in Figure 3.2

The grain boundaries were calculated by the 95% confidence limit procedure of ASTM E 112-88, and the volume fraction of recrystallized austenite was determined by standard point counting technique.

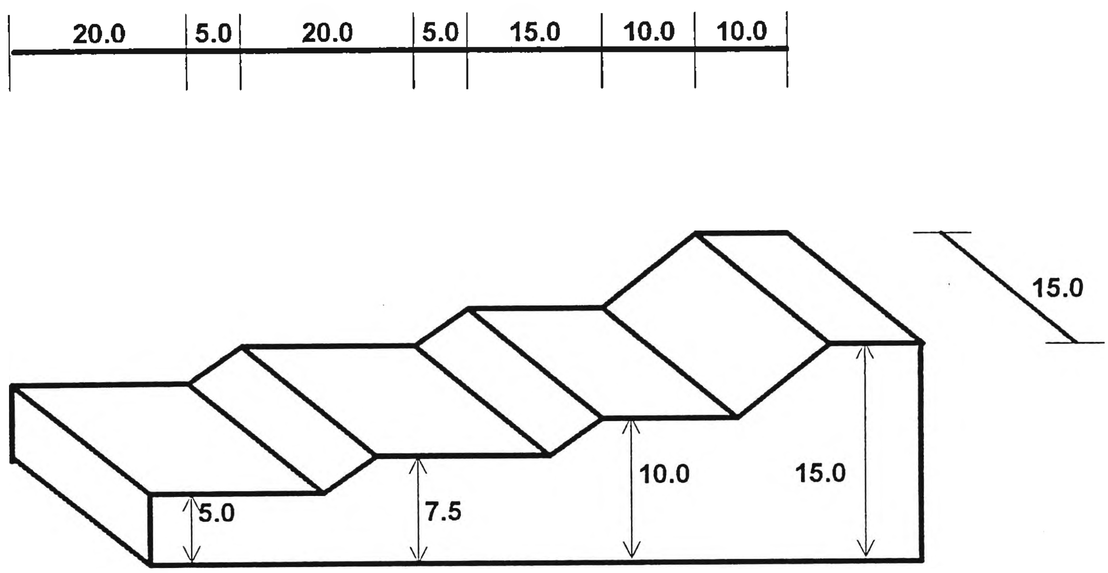

Figure 3.1. Dimension of Specimen Employed for Simultaneous Reductions Under Single Pass Rolling Conditions (mm).

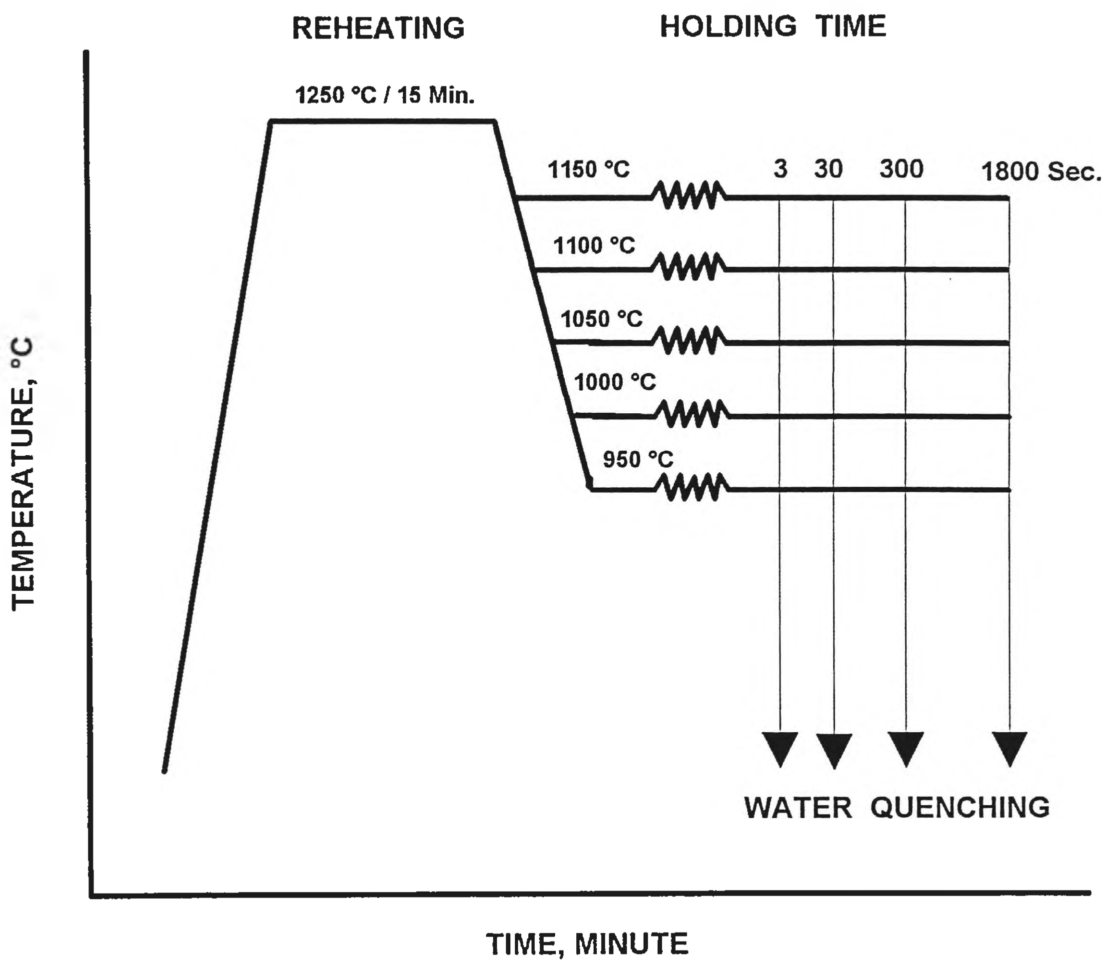

Figure 3.2. Rolling Schedule

CHAPTER 4

RESULTS

4. RESULTS

The three steels were investigated involved a reference C-Mn and two HSLA steels having different microalloying elements. The experiment were design to clarify the effects of variables of the rolling process such as temperature, reduction and holding time on the progress of recrystallization of austenite. The volume fraction of recrystallized austenite grain size and grain boundary area per unit volume were measured in quenched samples immediately after rolling and after also holding for up to 1800 sec.The quenching immediatelly after rolling took -3 sec, and can be regarded as being subjected to a holding time of 3 sec at temperature after rolling. This condition is referred to "as-quenched sample after deformation". The starting grain sizes (d_0) for these steels for subsequent hot rolling experiments are given in Table 4.1.

Table. 4.1. Grain Size In Steel (μm) After Heating To 1250°C For 15 Minutes

STEEL	% ELEMENT	INITIAL GRAIN SIZE (d_0) (μm)
C- Mn	-	290 ± 15
Nb - V	Nb : 0.03 V : 0.05	169 ± 10
Nb-V-Ti	Nb : 0.05 V : 0.03 Ti : 0.02	159 ± 8

4.1. GRAIN COARSENING BEHAVIOUR

The mean austenite grain size of the C-Mn steel increases progressivelly with an increase of reheating temperature, and from the results reported in the literature (24) the grain coarsening temperature of the C-Mn steel slab is about 950°C (Figure 4.1).

It is also clear that Nb-V and Nb-V-Ti steel increase the reheating temperature but only result in very slight increase in mean austenite grain size until grain coarsening occurs, which can be established as the grain coarsening temperature. And increasing temperature reheating continuosly resulted in the rapid increase in mean austenite grain size.

The grain coarsening temperature of the Nb-V steel is is higher than the grain coarsening temperature of C-Mn steel. The Nb-V-Ti steel has titanium content and which made grain coarsening temperature of Nb-V-Ti steel (1050°C) higher than for the C-Mn steel and the Nb-V steel.

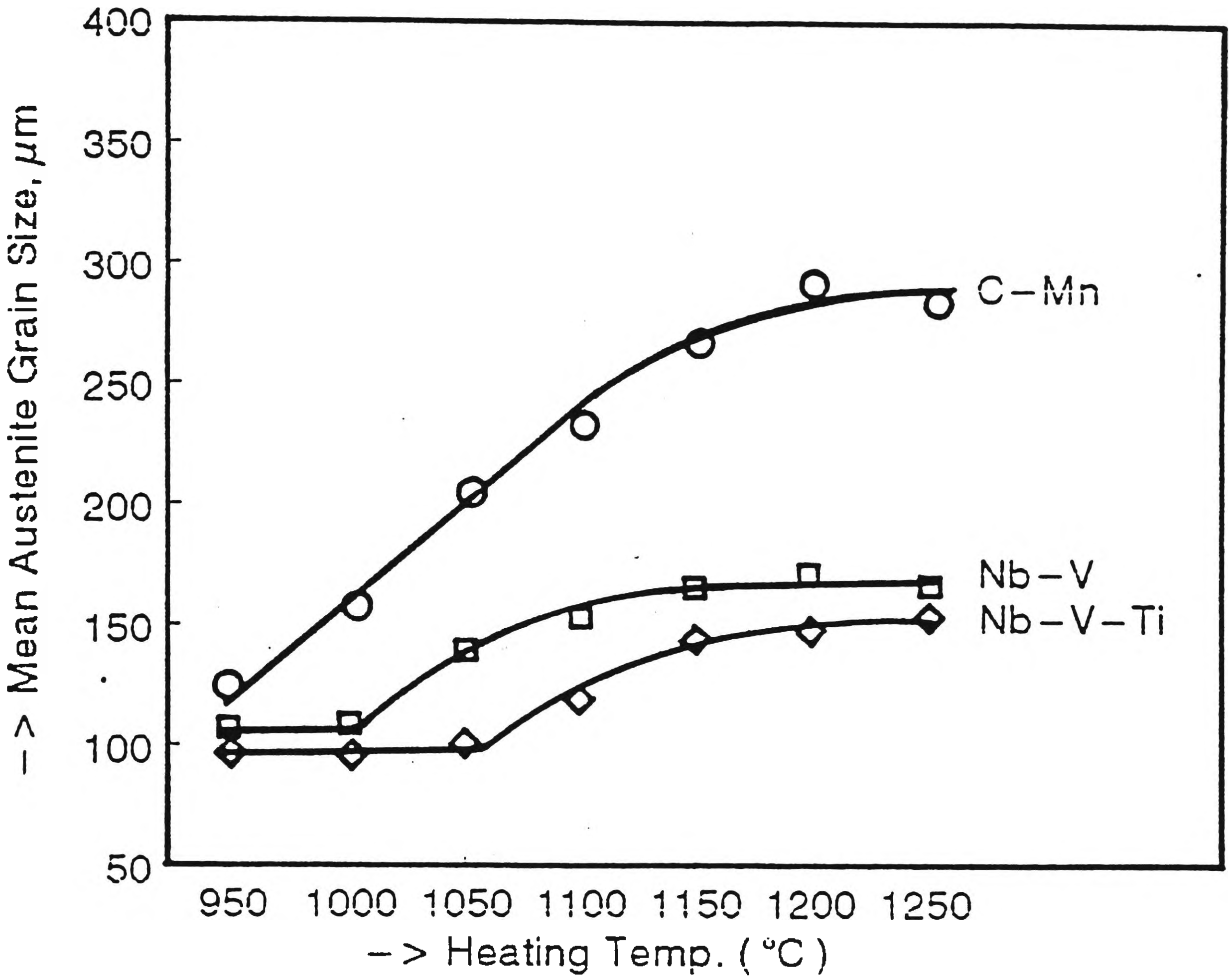

Figure 4.1. **Grain Coarsening Curve on C-Mn, Nb-V, and Nb-V-Ti Steels by Holding Time 15 minutes.**

4.2. AUSTENITE RECRYSTALLIZATION IN AS-QUENCHED STEEL

The effect of temperature and rolling reduction on recrystallization in the as-annealed plain carbon and microalloyed steels will now be discussed. The samples were quenched within 3 sec. after rolling and referred to "as-quenched conditon". The initial grain sizes of the steels were 290,169 and 159 μm for C-Mn, Nb-V and Nb-V-Ti steels, respectively, as shown in Table 4.1.

4.2.1. Effect of Rolling Temperature And Reduction

The effect of rolling temperature on the austenite recrystallization is shown in figures (4.2 - 4.4). From these figures It can be seen that for a given rolling reduction the amount of recrystallized austenite increases as deformation temperature increase for C-Mn, Nb-V and Nb-V-Ti steels.

For example, in the plain carbon steel a 40% reduction at 950°C resulted in 65% recrystallization, but at 1050°C the presentage of recrystallization was 95%. (samples quenched within 3 sec. after rolling).

Similarly if the deformation reduction increased to 60%, there was 90% recryatallization at 950°C and 95% recrystallization at 1000°C in plain carbon steel.

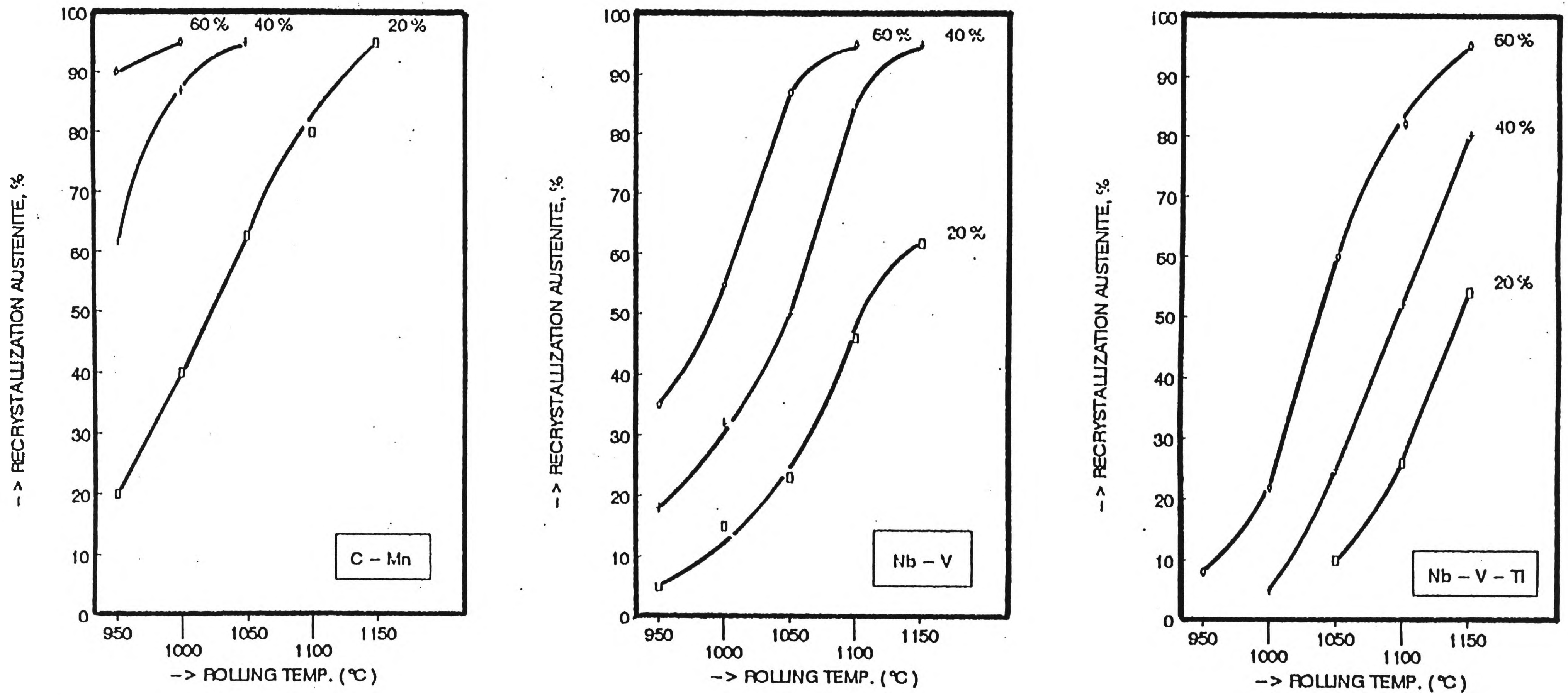

Figure 4.2. Effect of Rolling Temperature on Percent of Recrystallization in C-Mn, Nb-V, and Nb-V-Ti Steels, with Different reductions Samples Quenched within 3 sec. after Reheating at 1250°C for 15 minutes.

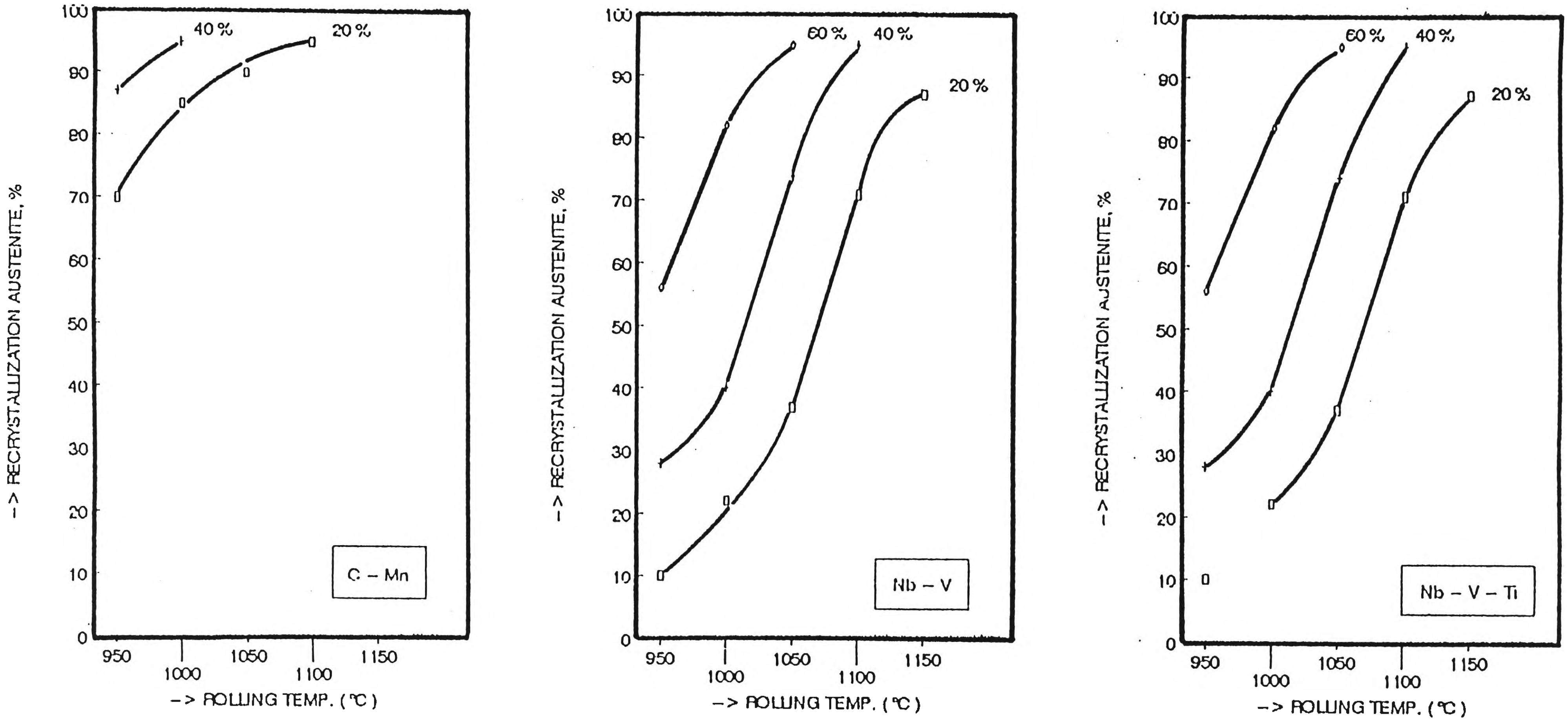

Figure 4.3. Effect of Rolling Temperature on Percent of Recrystallization in C-Mn, Nb-V, and Nb-V-Ti Steel, with Different Reduction Samples Quenched within 30 sec. after Reheating at 1250°C for 15 minutes.

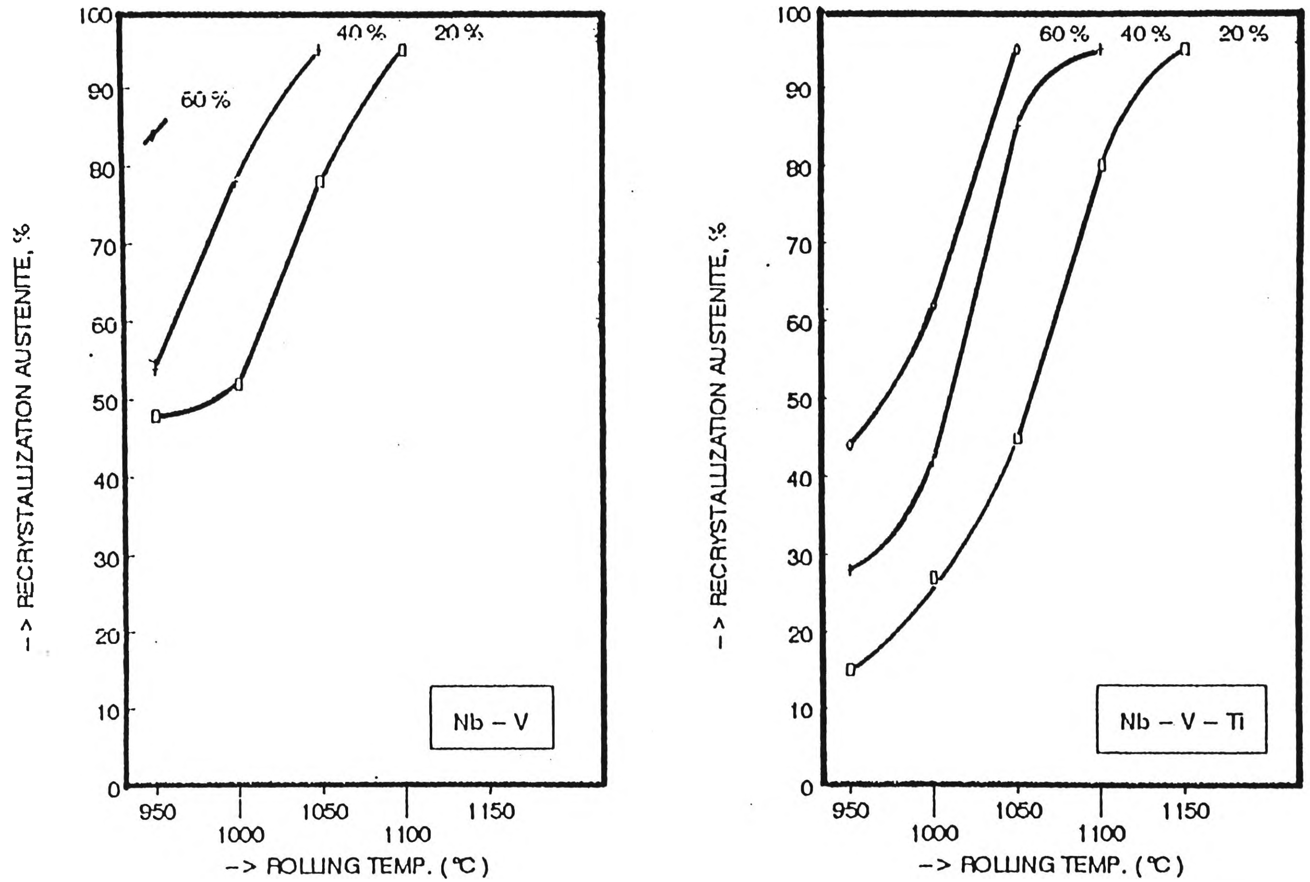

Figure 4.4. Effect of Rolling Temperature on Percent of Recrystallization in C-Mn, Nb-V, and Nb-V-Ti Steel, with Different Reduction Samples Quenched within 300 sec. after Reheating at 1250°C for 15 minutes.

Presence of microalloying elements in Nb-V and Nb-V-Ti steels showed such behaviour in which the increase in the amount of reduction or an increase temperature, increased the proportion of recrystallized austenite grain, except that proportion of recrystallized austenite was lower in microalloyed steel compared to C-Mn steel under all conditions.

Photomicrographs in Figures 4.5 - 4.7 show austenite grain structure for direct quenched (3 sec.) samples at temperature 1050°C for Nb-V steel and at 950°C and 1150°C for Nb-V-Ti steel with 20%,40% and 60% reduction.

4.2.2. Effect Of Holding Time

The effect of holding time on the austnite recrystallization increased the proportion of austenite recrystallized as shown in figures 4.8.

After 40% rolling, the times needed for 95% recrystallization at 1050°C for C-Mn, Nb-V and Nb-V-Ti steels were 3 sec., 300 sec. and 1800 sec., respectively. At temperature of 950°C, with rolling at 40% reduction and holding within 3 sec., the percentage of recrystallized austenite grains was 62% for C-Mn, but the Nb-V steel was only 18% recrystallized and the Nb-V-Ti steel was completely unrecrystallized. It can be seen from Figure 4.8, that for the C-Mn steel reduced 40% at 950°C, the volume fraction of recrystallized austenite increased from 62% to 95% when the holding time increased from 3 sec. to 300 sec. And the volume fraction of recrystallized austenite in the Nb-V steel increase from 18% to 95% when the holding time increased from 3 sec. to 1800 sec. On the other hand, in the Nb-V-Ti steel recrystallization strarted after 30 sec. and progressed to only 84% after 1800 sec. Comparison of the C-Mn with the two microalloyed steels at the same reduction indicates that recrystallization time is greater in the microalloyed steel than in the C-Mn steel. The time of holding necessary to complate recrystallization decreases with increase in temperature or reduction. Photomicrographs in Figure 4.9. show the austenite grain structure in C-Mn steel with holding time 300 sec. at 1150°C rolling temperature, and Figures 4.10 and 4.11 show the austenite grain structure in the Nb-V steel with holding time 300 sec. and 1800 sec. at 950°C rolling temperature.

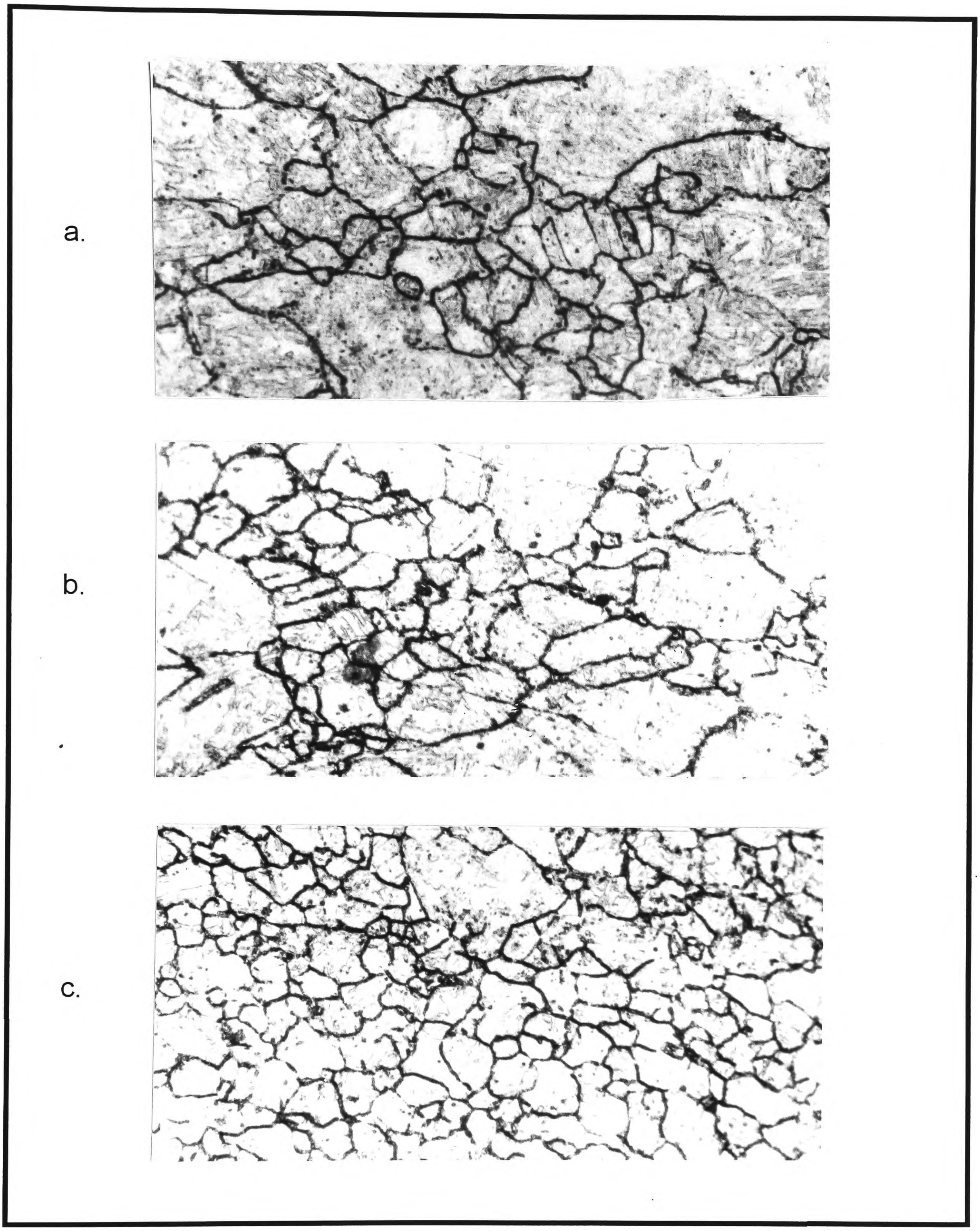

Figure 4.5. Photomicrographs Showing Austenite Grain Structure in Direct Quenched Nb-V Steel After 15 Minutes at 1250°C (d_0 = 169 μm); (a) 20% Reduction at 1050°C, (b) 40% Reduction at 1050°C, and (c) 60% Reduction at 1050°C. Etched in Aqueous Picric Acid. 200 x.

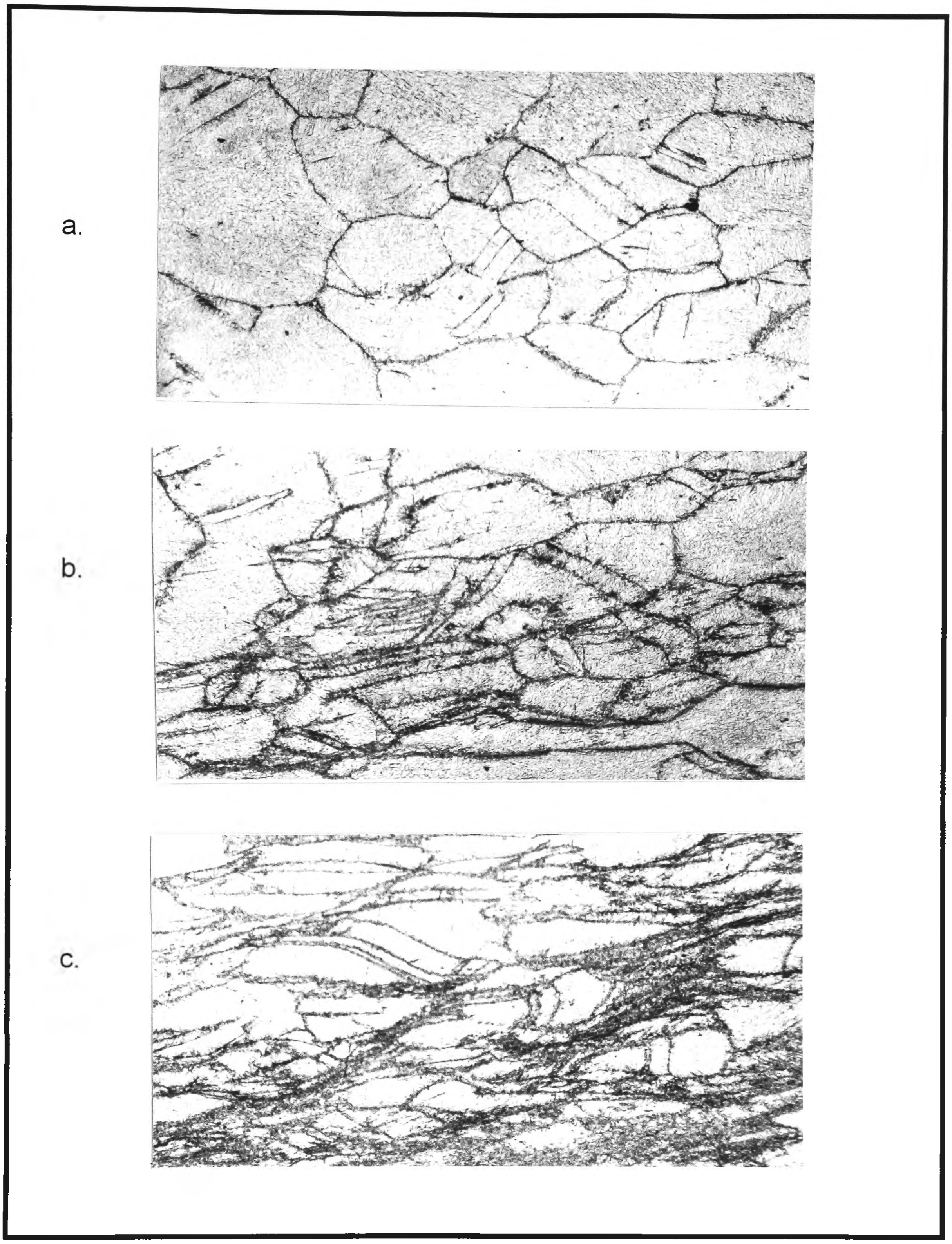

Figure 4.6. Photomicrographs Showing Austenite Grain Structure in Direct Quenched Nb-V-Ti Steel After 15 Minutes at 1250°C (d_0 = 159 μm); (a) 20% Reduction at 950°C, (b) 40% Reduction at 950°C, and (c) 60% Reduction at 950°C. Etched in Aqueous Picric Acid. 200 x.

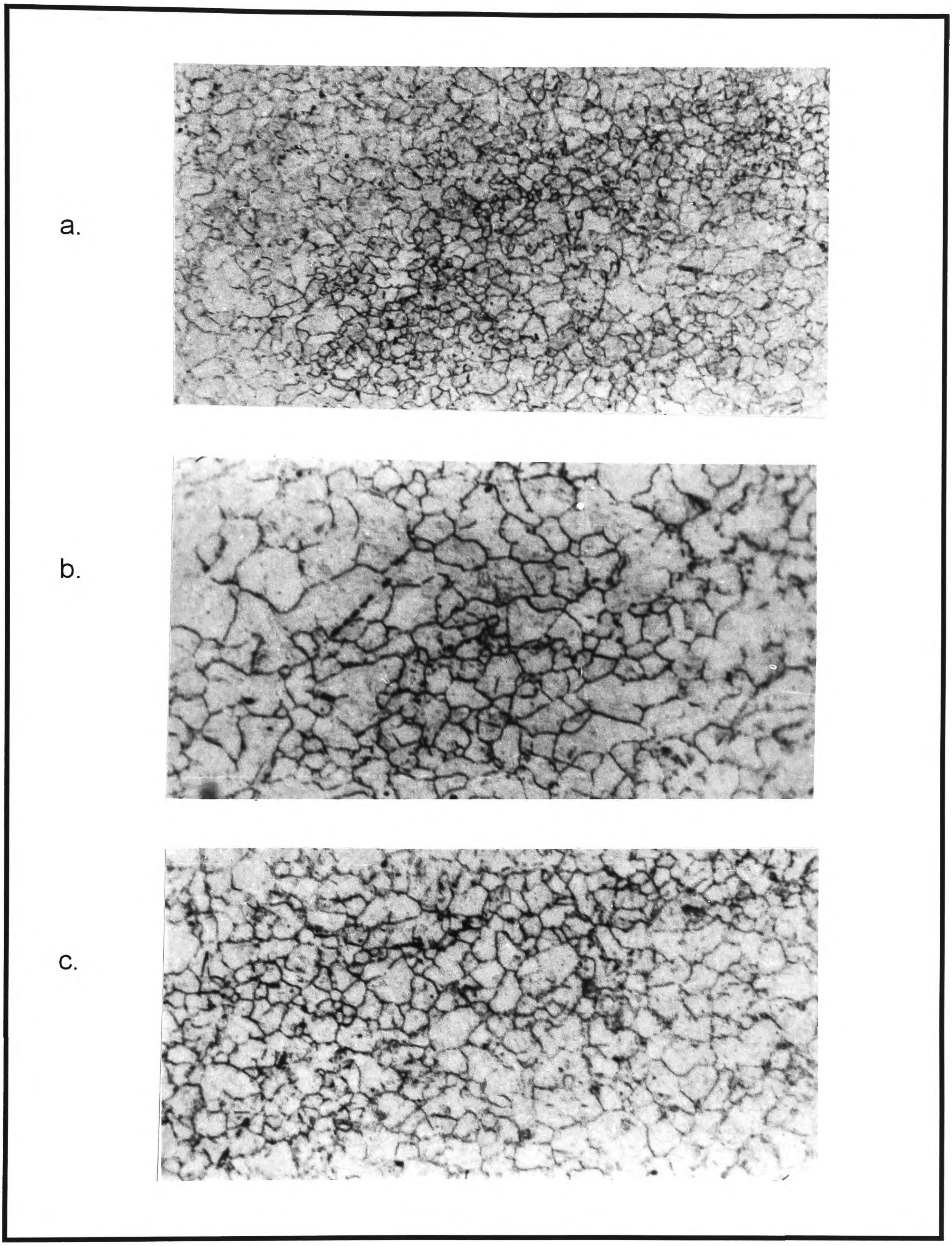

Figure 4.7. Photomicrographs Showing Austenite Grain Structure in Direct Quenched Nb-V-Ti Steel After 15 Minutes at 1250°C (d_0 = 159 μm); (a) 20% Reduction at 1150°C, (b) 40% Reduction at 1150°C, and (c) 60% Reduction at 1150°C. Etched in Aqueous Picric Acid. 200 x.

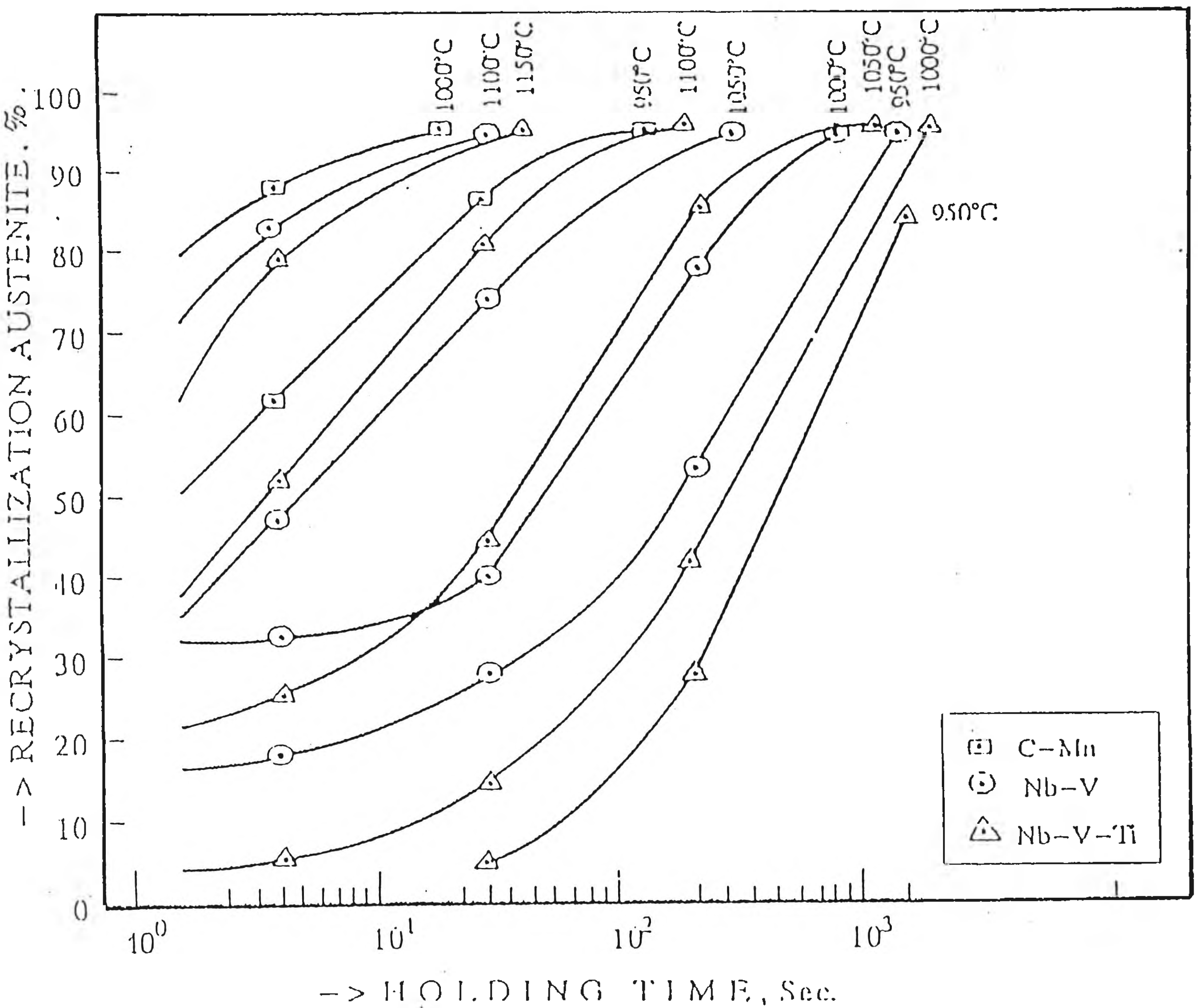

Figure 4.8. Effect of Holding Time in C-Mn, Nb-V and Nb-V-Ti Steels on Recrystallization, for 40% Reduction and Different Rolling Temperature.

a.

b.

c.

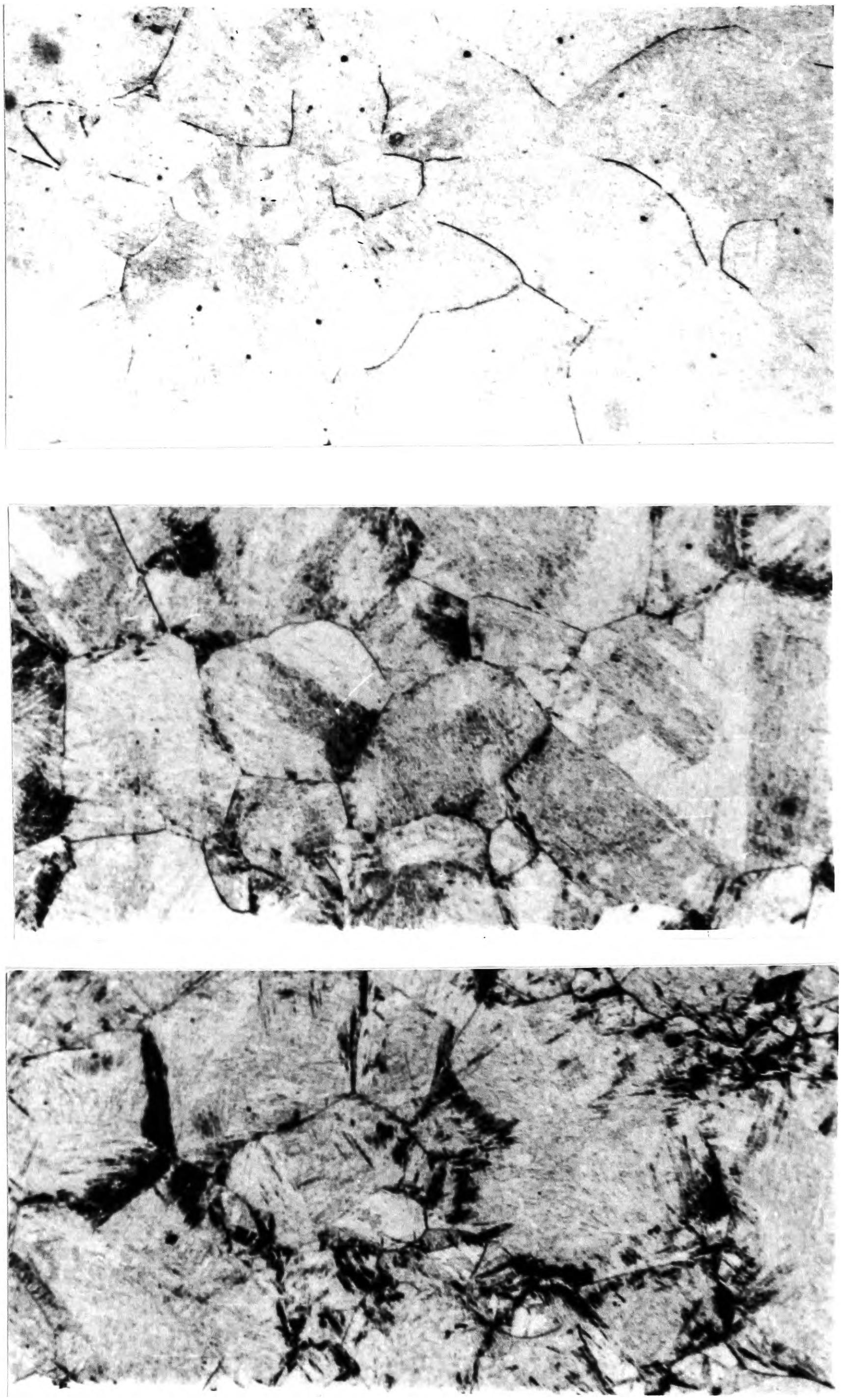

Figure 4.9. **Photomicrographs Showing Austenite Grain Structure in C-Mn Steel After 15 Minutes at 1250°C (d_0 = 269 μm) with Holding Time 300 sec. (a) 20% Reduction (b) 40% Reduction and (c) 60% Reduction at 1150°C. Etched in Aqueous Picric Acid. (a) 200 x & (b), (c) 100 x.**

a.

b.

c.

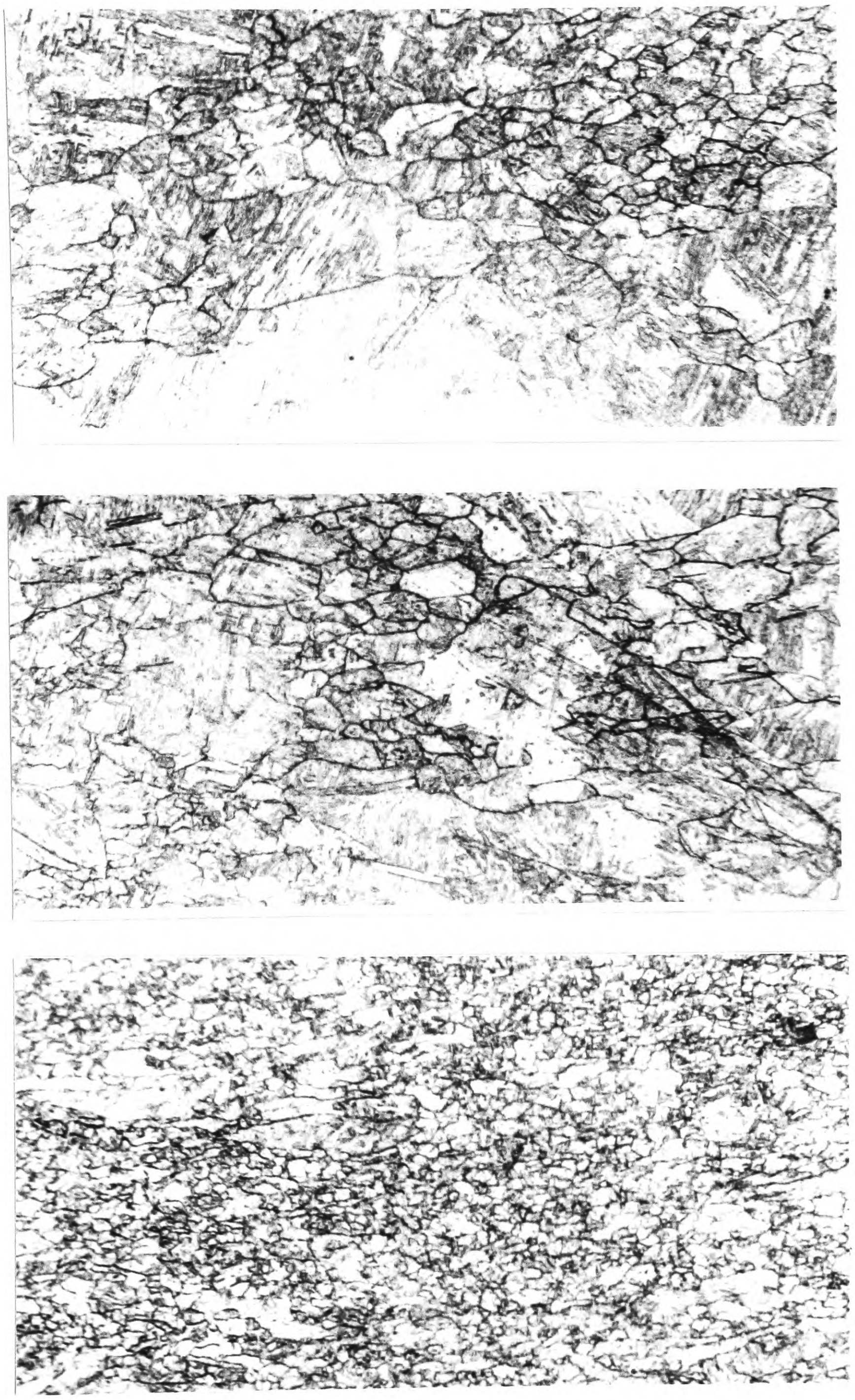

Figure 4.10. Photomicrographs Showing Austenite Grain Structure in Nb-V Steel After 15 Minutes at 1250°C (d_0 = 169 μm); (a) 20% Reduction (b) 40% Reduction and (c) 60% Reduction at 950°C and Holding Time 300 sec.. Etched in Aqueous Picric Acid. (a), (b) & (c) 100 x.

a.

b.

c.

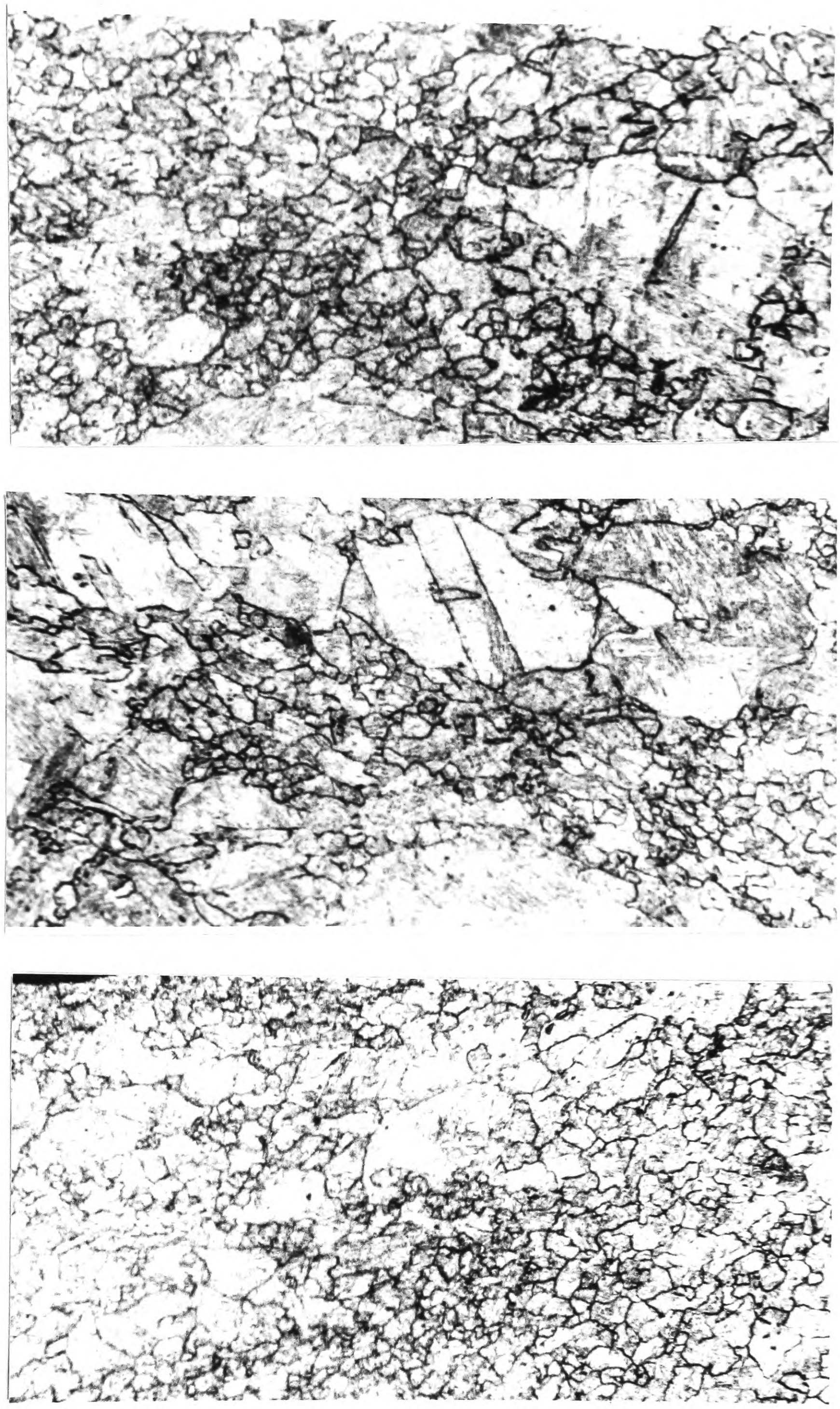

Figure 4.11. Photomicrographs Showing Austenite Grain Structure in Nb-V Steel After 15 Minutes at 1250°C (d_0 = 169 μm); (a) 20% Reduction (b) 40% Reduction and (c) 60% Reduction at 950°C, and Holding Time 1800 sec.. Etched in Aqueous Picric Acid. 100 x.

4.3. PROGRESS OF AUSTENITE RECRYSTALLIZATION IN C-Mn, Nb-V AND Nb-V-Ti STEELS.

The effect of rolling temperature and % reduction on the state of austenite recrystallization for the C-Mn, Nb-V and Nb-V-Ti steel is given in Figures 4.12 - 4.14. The austenite in the non recrystallized state (NR) is indicated as the open circles and the line RS (recrystallization start) indicates the critical reduction necessary to initiate recrystallization within 3 sec. after rolling at various temperatures.

The microstructure consists of recystallized and non-recrystallized austenite grain indicated by the PR (partial recrystallization) curve represented by half-filled circles. The amount of austenite recrystallized austenite increases with an increase in the reduction and temperature of deformation, and recrystallization reaches to completion at the critical reduction and temperature combination as indicated by the RF (fully recrystallization), curve as shown in Figure 4.12 The non recrystallization (NR), partial recrystallization (PR) and recryatallization (R) regions are separated by the RS and RF curves. In the recrystallization region, numbers in circles represent austenite recrystallized grain size measured in microns.

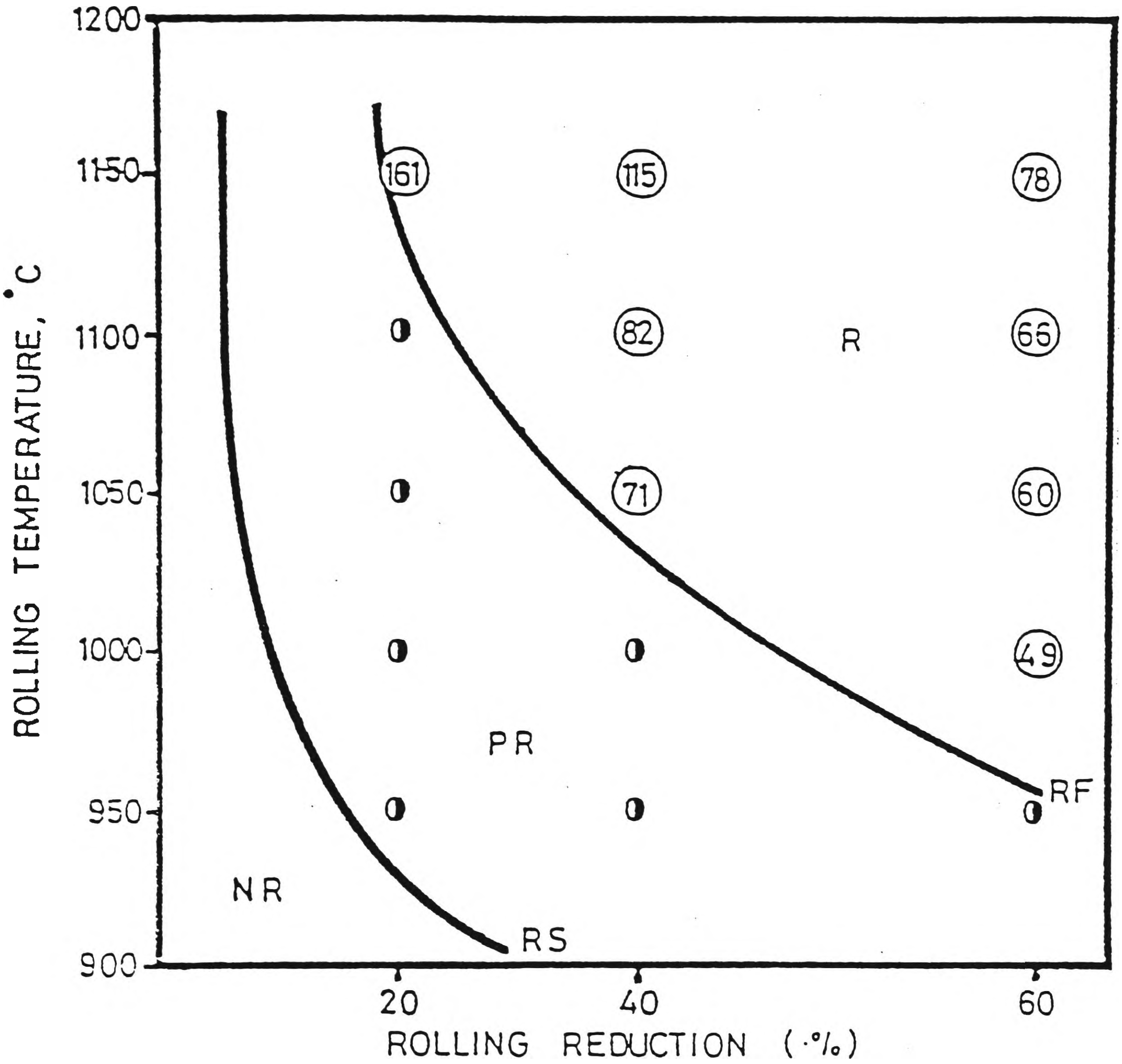

Figure 4.12. Austenite Recrystallization as a Function of Rolling Temperature and Reduction in C-Mn Steel and Resulting Grain Size Figures in Circles Show Recrystallized Grain Size (μm). The Samples were Reheated to 1250°C and Rolled with One Pass and Quenched within 3 Sec.

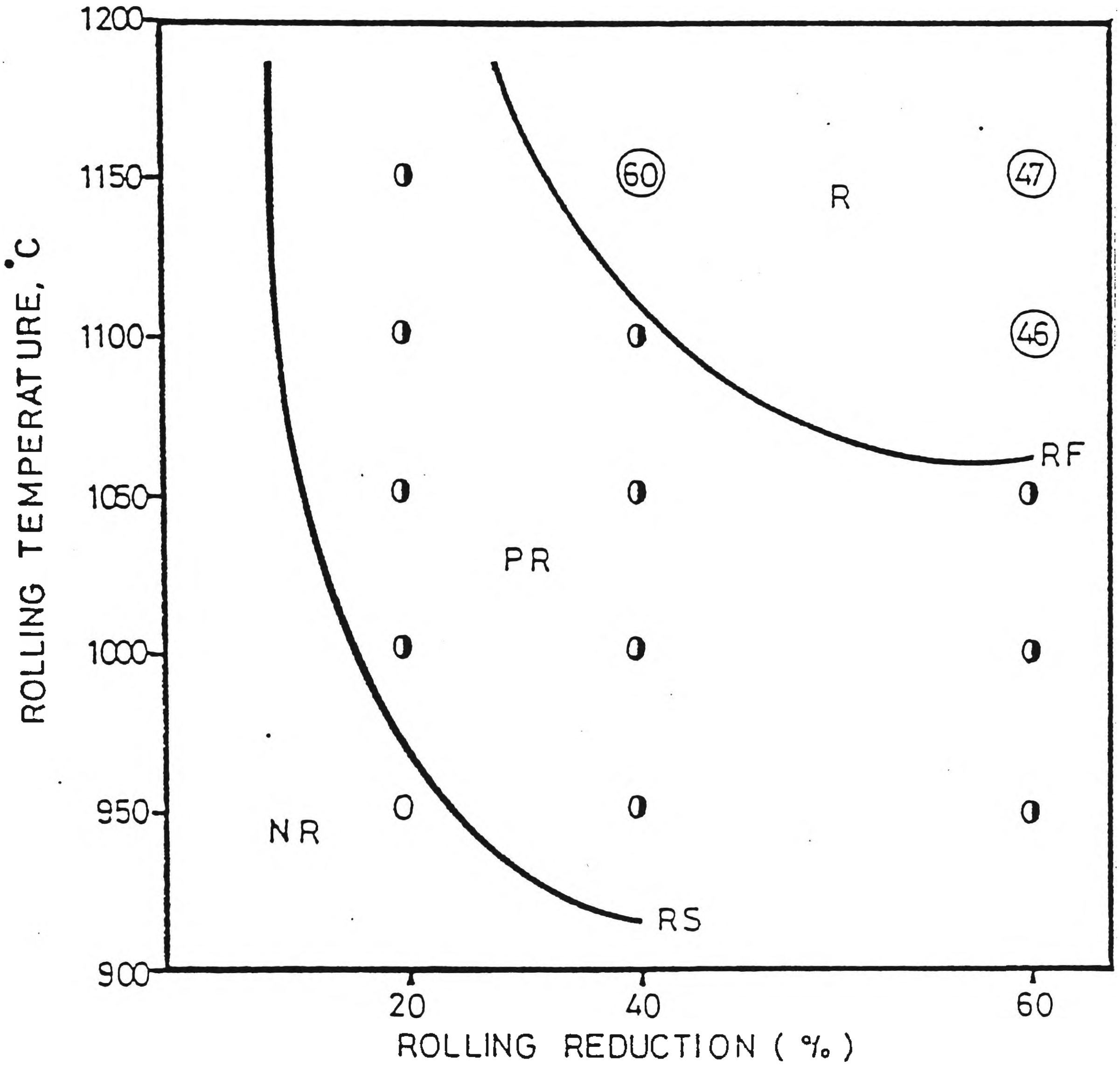

Figure 4.13 . **Austenite Recrystallization as a Function of Rolling Temperature and Reduction in Nb-V Steel and Resulting Grain Size Figures in Circles Show Recrystallized Grain Size (μm). The Samples were Reheated to 1250°C and Rolled with One Pass and Quenched within 3 Sec.**

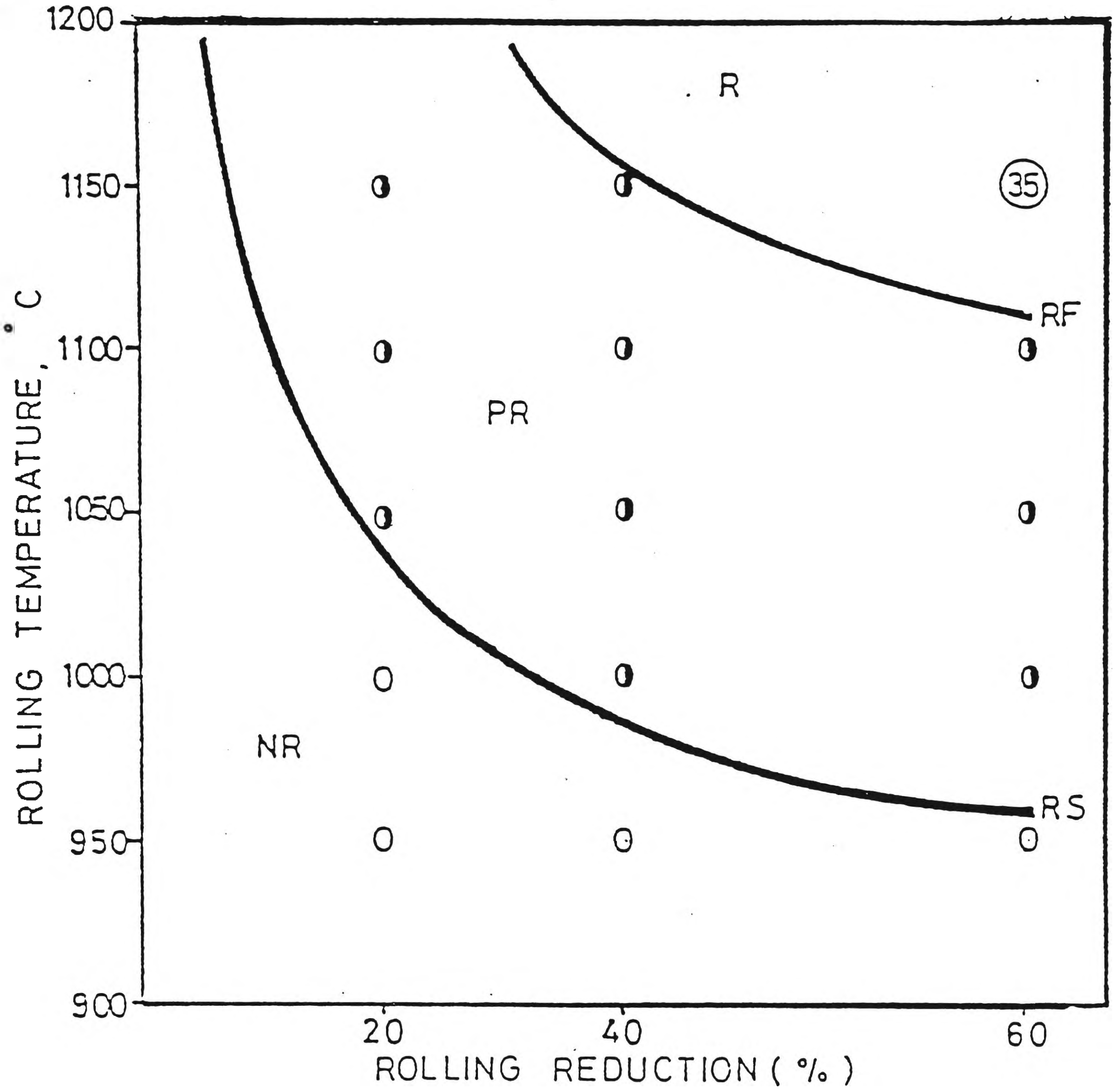

Figure 4.14 . Austenite Recrystallization as a Function of Rolling Temperature and Reduction in Nb-V-Ti Steel and Resulting Grain Size Figure in Circles Show Recrystallized Grain Size (μm). The Samples were Reheated to 1250°C and Rolled with One Pass and Quenched within 3 Sec.

4.3.1. Effect Of Rolling Temperature

The mean austenite grain size vs temperature curves obtained on direct quenching after 20%, 40% and 60% reductions to samples of C-Mn, Nb-V and Nb-V-Ti steels are shown in Figure 4.15 - 4.18. The trend in grain size in these cases should be falling from the starting grain size to a minimum then an upward trend with either holding time at constant temperature, or with holding temperature at constant time, due to coarsening. However, for clarify the falling lines have been omitted. From the figures it can be seen that the grain size increases with increase in temperature of rolling. In Nb-V and Nb-V-Ti samples, increasing the rolling temperature from 950°C to 1050°C increased the grain size slightly in 40% and 60% rolling condition indicating that austenite recrystallization had just begun (Figure 4.15).

When the temperature was increased from 1050°C up to 1100°C, normal grain growth took place in the Nb-V and the Nb-V-Ti steels. In the C-Mn steel samples, increasing the rolling temperature for 950°C to 1150°C increased the grain size rapidly in 40% and 60% rolling condition indicating that complete recrystallization is reached. The onset of recrystallization required a higher temperature, the initial grain size was lauger in the C-Mn and was smaller in the Nb-V and the Nb-V-Ti steels and showing the refining effect of microalloy elements.

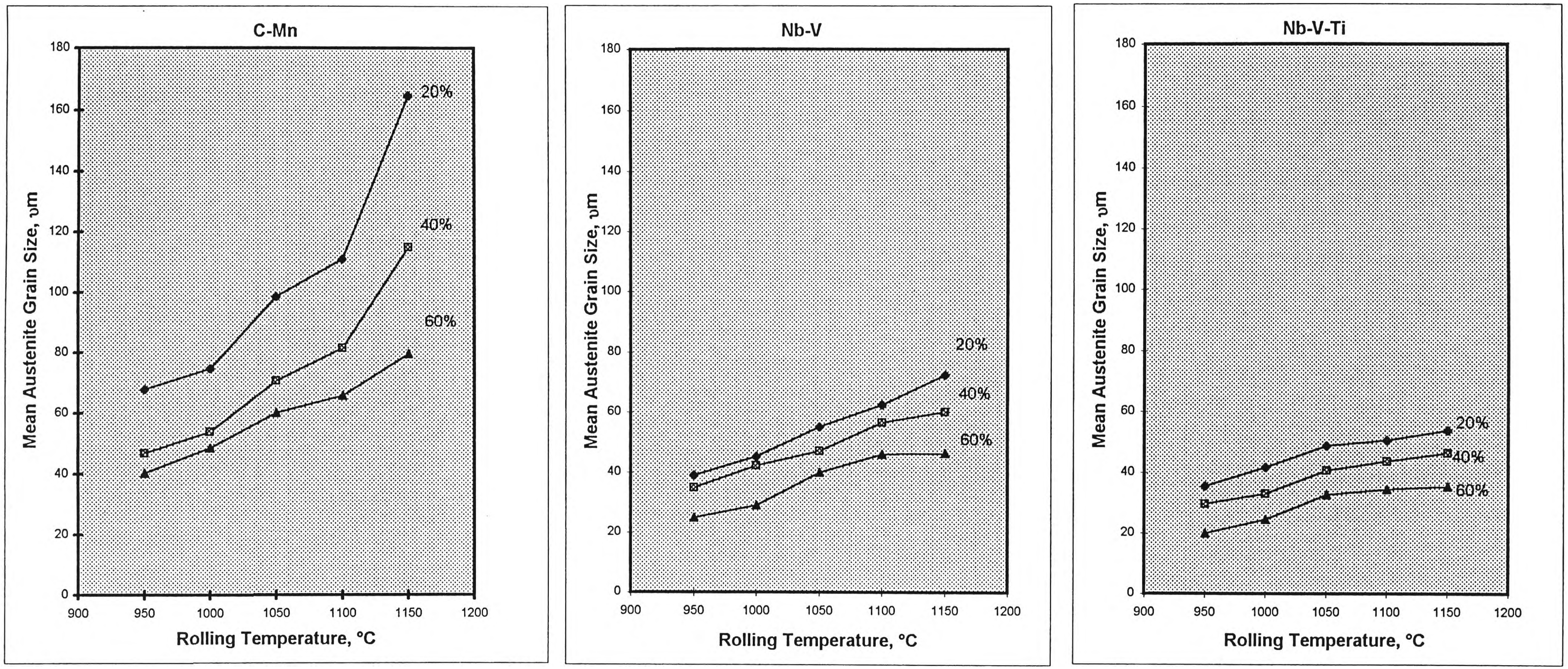

Figure 4.15 Effect of rolling temperature (°C) on mean austenite grain size for 20%, 40% and 60% reduction, samples quenched within 3 sec. after deformation of C-Mn, Nb-V and Nb-V-Ti steel.

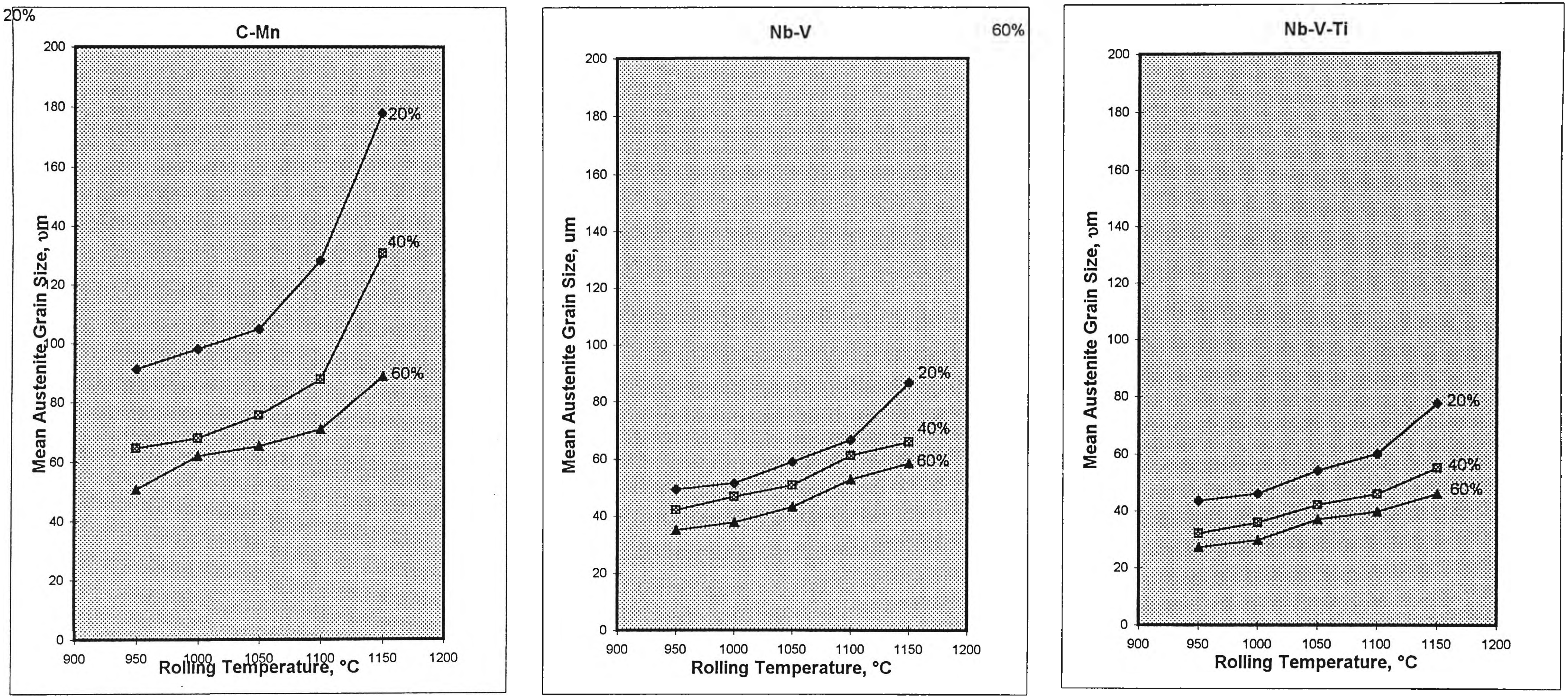

Figure 4.16 Effect of rolling temperature (°C) for mean austenite grain size of 20%, 40% and 60% reduction, samples quenched within 30 sec. after deformation of C-Mn, Nb-V and Nb-V-Ti steel.

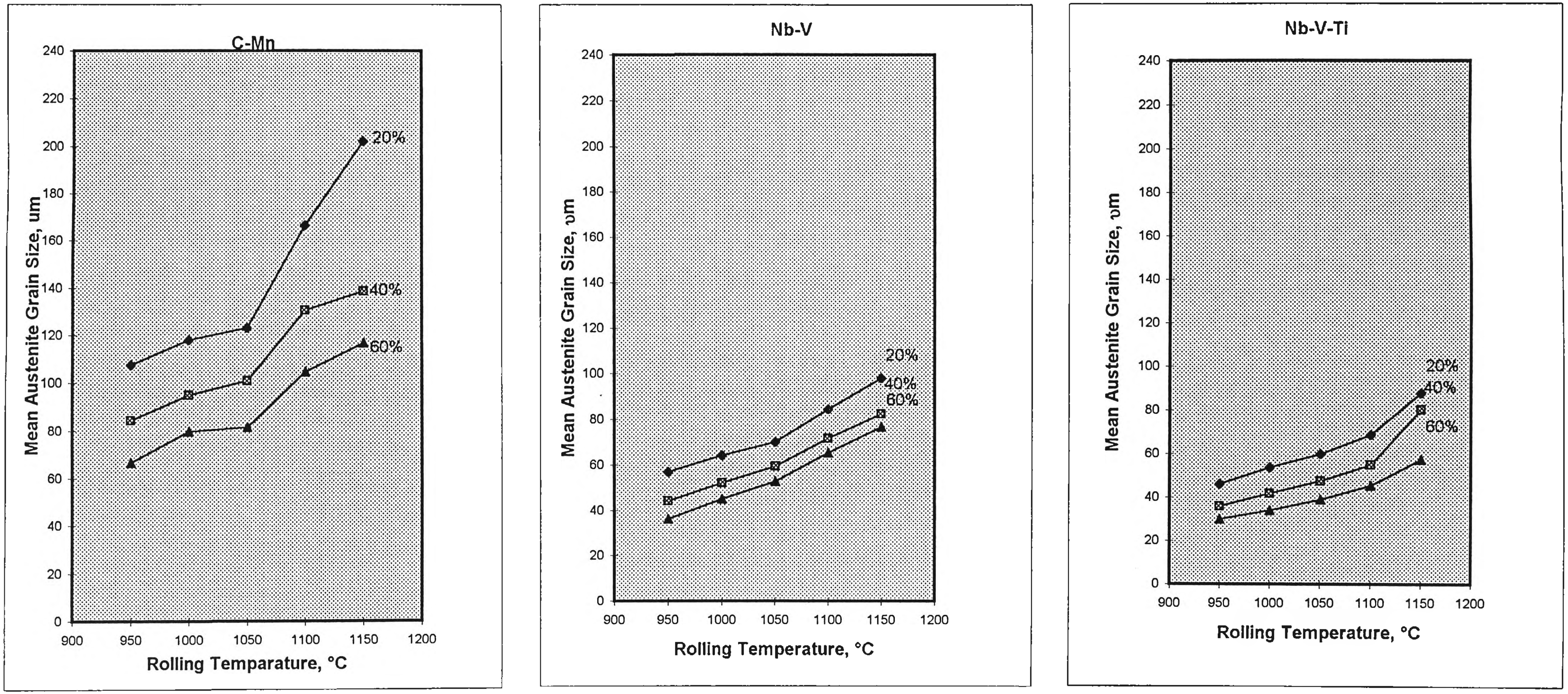

Figure 4.17 Effect of rolling temperature (°C) for mean austenite grain size of 20%, 40% and 60% reduction, samples quenched within 300 sec. after deformation of C-Mn, Nb-V and Nb-V-Ti steel.

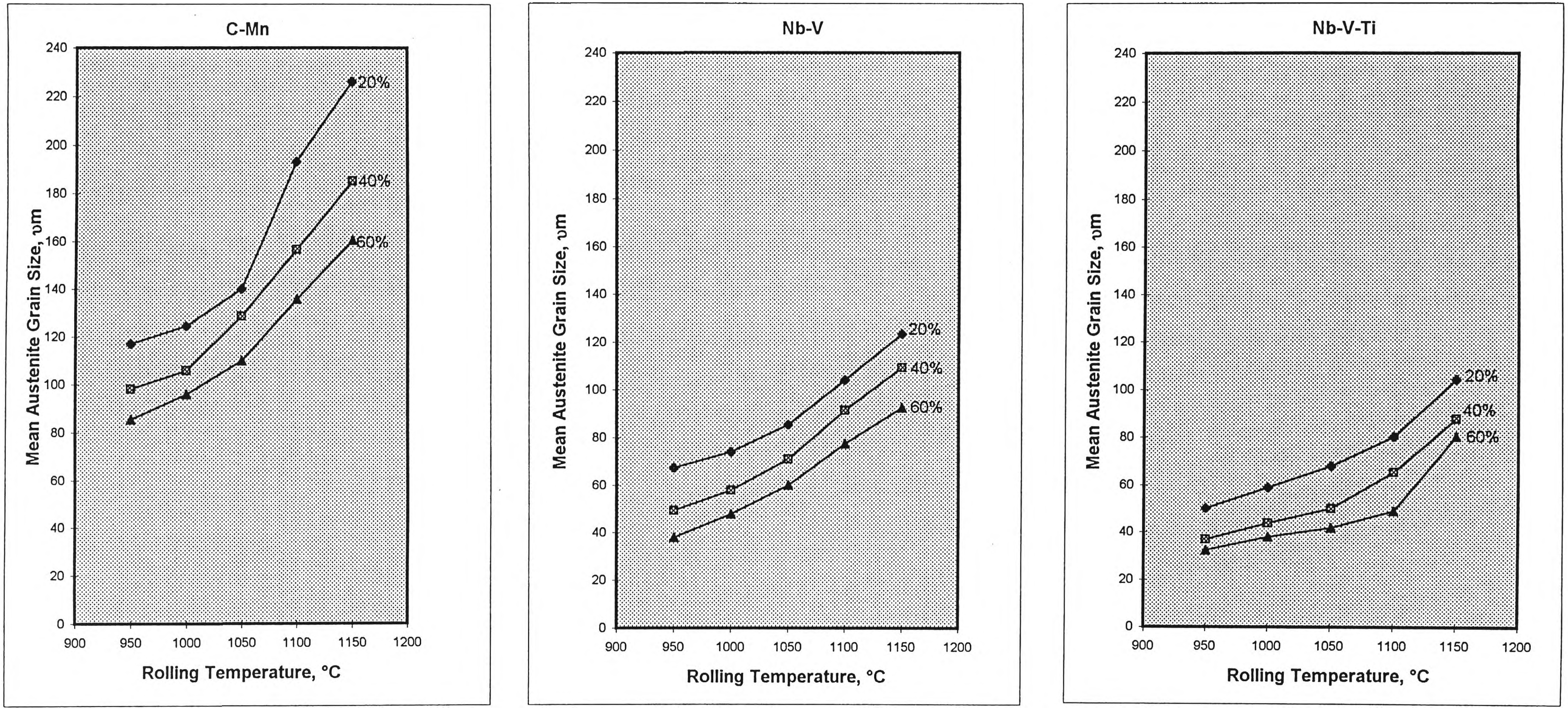

Figure 4.18 Effect of rolling temperature (°C) for mean austenite grain size of 20%, 40% and 60% reduction, samples quenched within 1800 sec. after deformation of C-Mn, Nb-V and Nb-V-Ti steel.

4.3.2. Effect Of Deformation

Figures (4.19 - 4.21) show the effect of amount of reduction on mean austenite grain size in direct quenched samples after rolling.

The curves in Figure 4.19 for various rolling temperature at reduction 20%, 40% and 60%, indicate the mean austenite grain size decreases in reduction. The mean grain size vs rolling reduction curves at various temperatures (Figure4.19 - 4.21) for the C-Mn compared to the Nb-V and the Nb-V- steels, show the grain size decrease similarly with increase in the percentage reduction for both lower and higher starting grain size. The Nb-V and the Nb-V-Ti steels with d_0 = 159 μm and $d_{0,}$ = 159 μm, the effect of reduction in fine grained steel is similar to that in samples the C-Mn steel with large initial grain size (d_0 = 290 μm), and continuously decrease in grain size with increase in reduction.

However, the recrystallized grains were much finer for the smaller initial grain size. For example, 60% reduction of samples at 1050°C resulted in mean recrystallized grain sizes of 32 μm and 60 μm in the Nb-V-Ti steel (d_0 = 159 μm) and the C-Mn steel (d_0 290 μm), respectively.

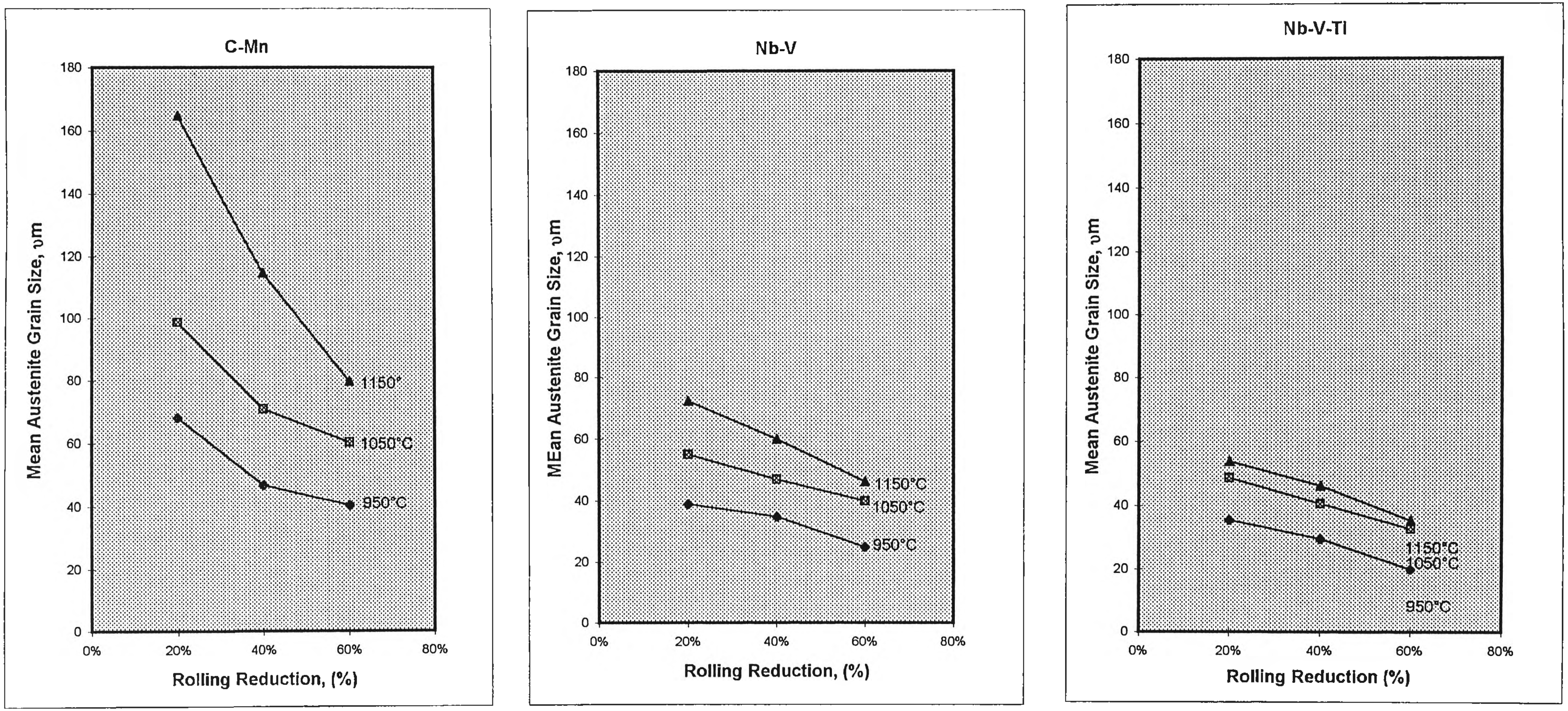

Figure 4.19 Effect of rolling reduction (%) on mean austenite grain size for different rolling temperature, samples quenched within 3 sec. after deformation of C-Mn, Nb-V and Nb-V-Ti steel.

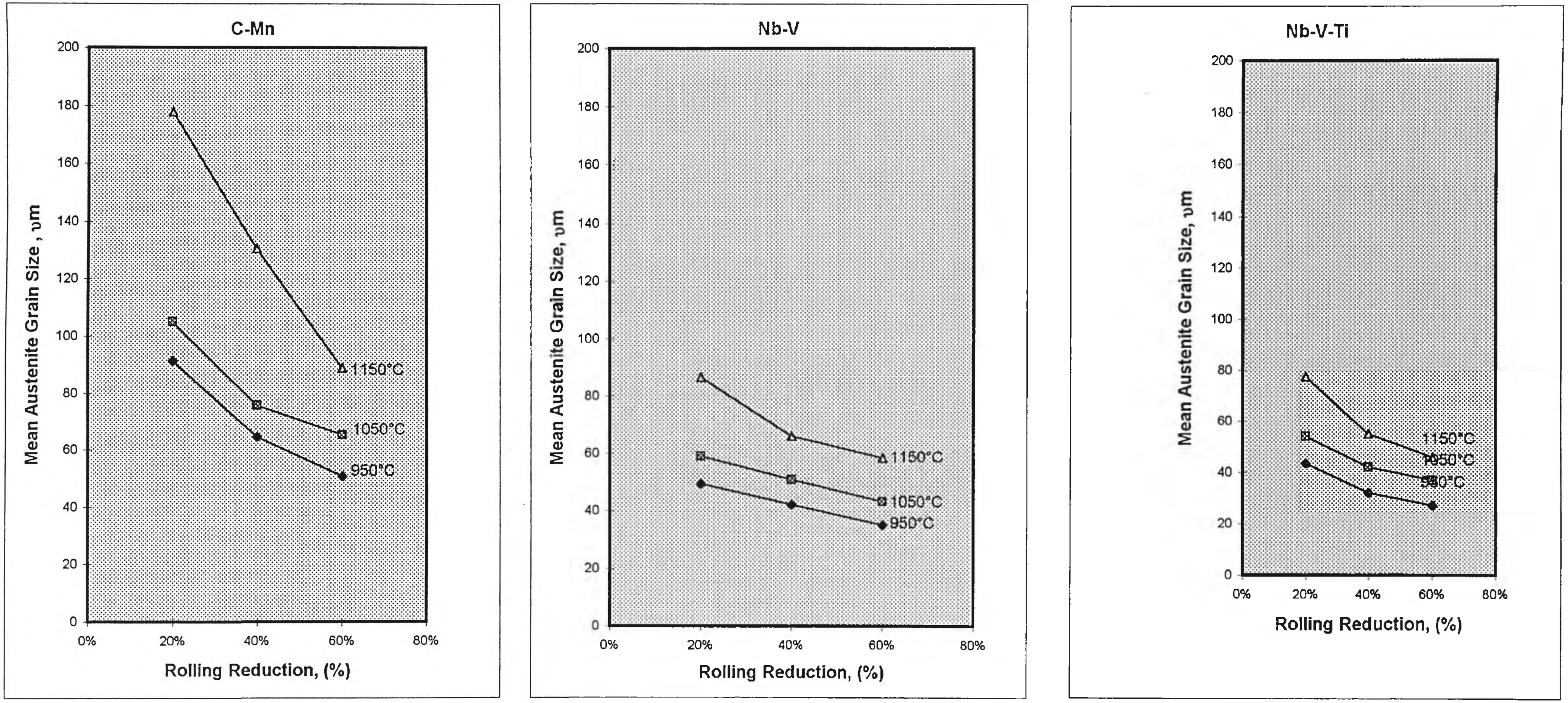

Figure 4.20 Effect of rolling reduction (%) on mean austenite grain size for different rolling temperature, samples quenched within 30 sec. after deformation of C-Mn, Nb-V and Nb-V-Ti steel.

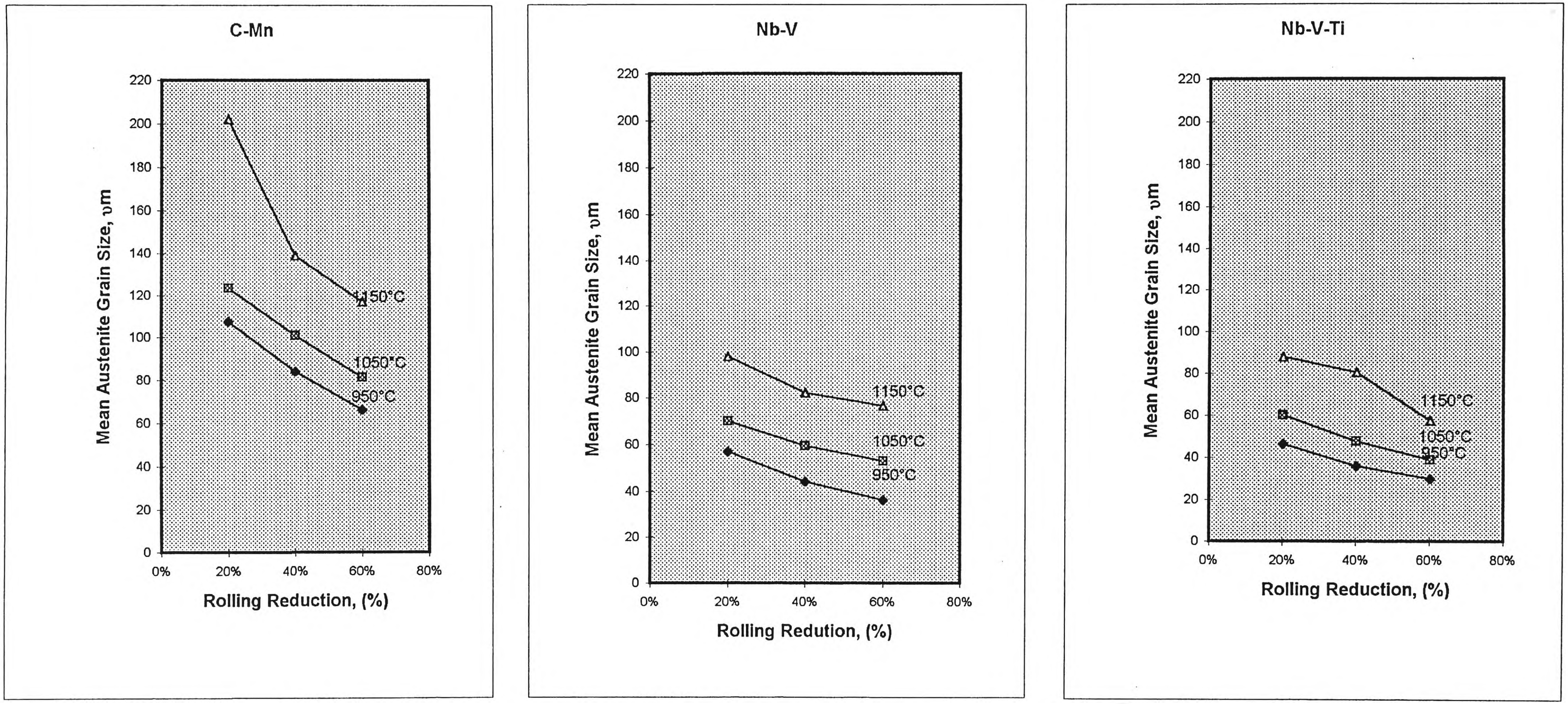

Figure 4.21 Effect of rolling reduction (%) on mean austenite grain size for different rolling temperature, samples quenched within 300 sec. after deformation of C-Mn, Nb-V and Nb-V-Ti steel.

4.3.3. Effect Of Holding Time

The effect of holding time on mean austenite grain size for different reductions and rolling temperature in the C-Mn, Nb-V and Nb-V-Ti steels is shown in Figure 4.22 - 4.26. If the rolling condition correspounds to a non- recrystallized region, the mean austenite grain size very slightly increased from initial grain size as the holding time increased. When the holding time increased beyond the non-recrystallization region, un-recrystallize austenite grain start to recrystallize simultaneously with grain growth. For example, the Nb-V and the Nb-V-Ti steels samples rolled 60% at 950°C (Figure 4.22) showed that mean grain size was 25 μm to 38 μm and 20 μm to 32 μm, respectively for holding times up to 1800 sec. In the C-Mn, the rate of increase in grain size with time was generally higher than for the Nb-V and Nb-V-Ti steels. This is readily understood because of the expected grain growth occuring simultaneously with, and after the completion of, recrystallization.

After the critical time for complete recrystallization is reached, fully recrystallized equiaxed grains result. Further holding of the sample at the rolling temperature produce grain growth.

When the rolling conditions result in recrystallization, the mean austenite grain size increased for rapidly holding time increase, and this can be seen at higher reduction (60%). This point is illustrated by the Nb-V and the Nb-V-Ti steels reduced 60% at 1050°C and 1100°C. (Figure4.24 - 4.25).

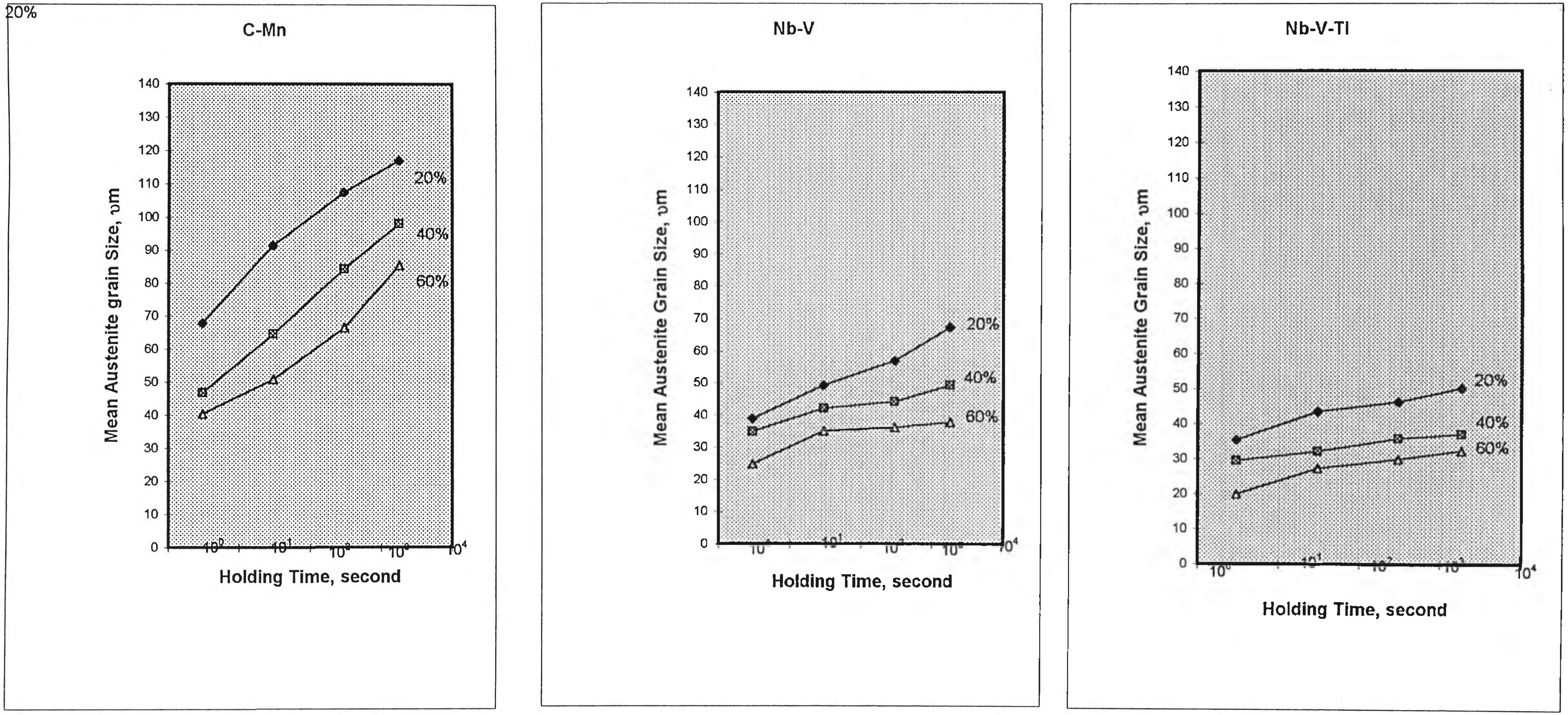

Figure 4.22 Effect of holding time(second) on mean austenite grain size for different reduction, at rolling temperature 950°C of C-Mn, Nb-V and Nb-V-Ti steel.

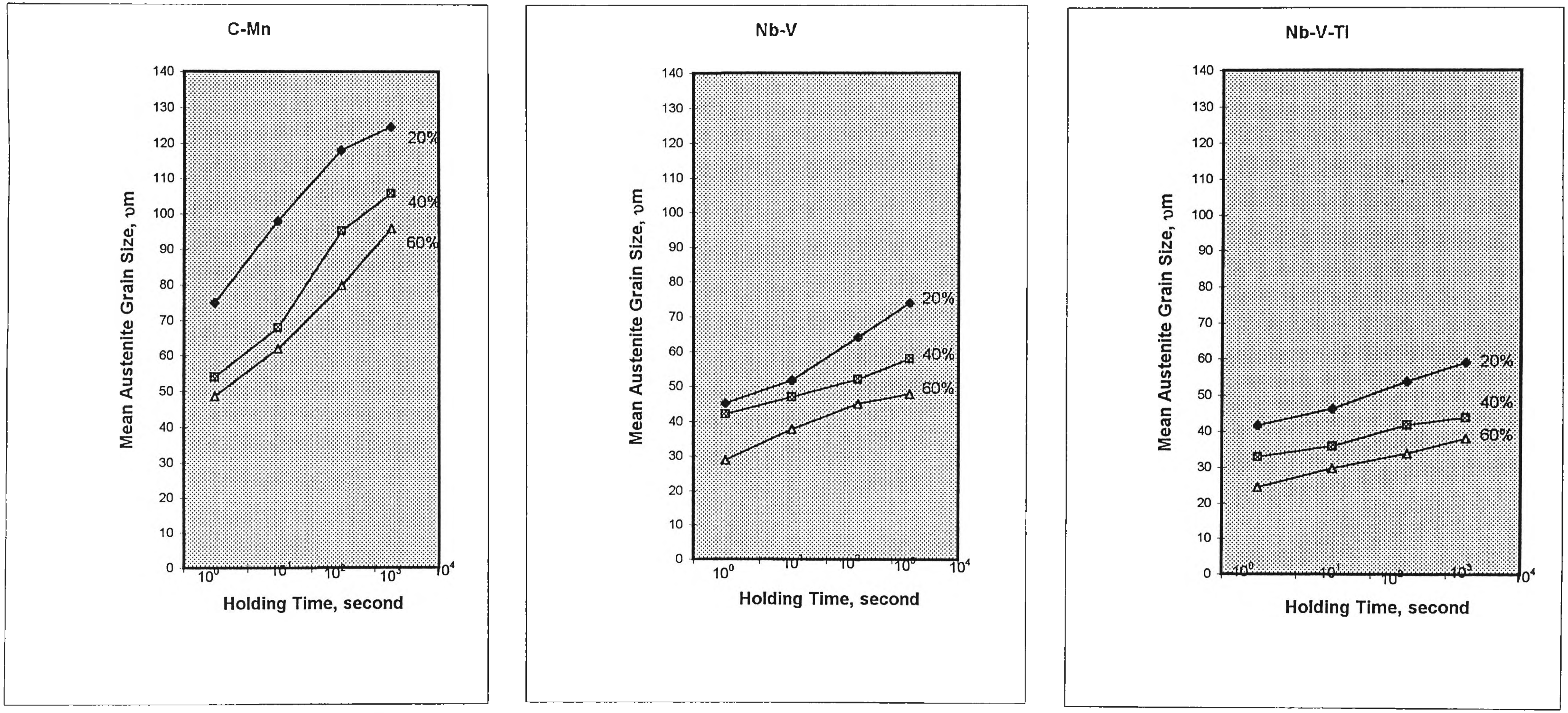

Figure 4.23 Effect of holding time(second) on mean austenite grain size for different reduction, at rolling temperature 1000°C of C-Mn, Nb-V and Nb-V-Ti steel.

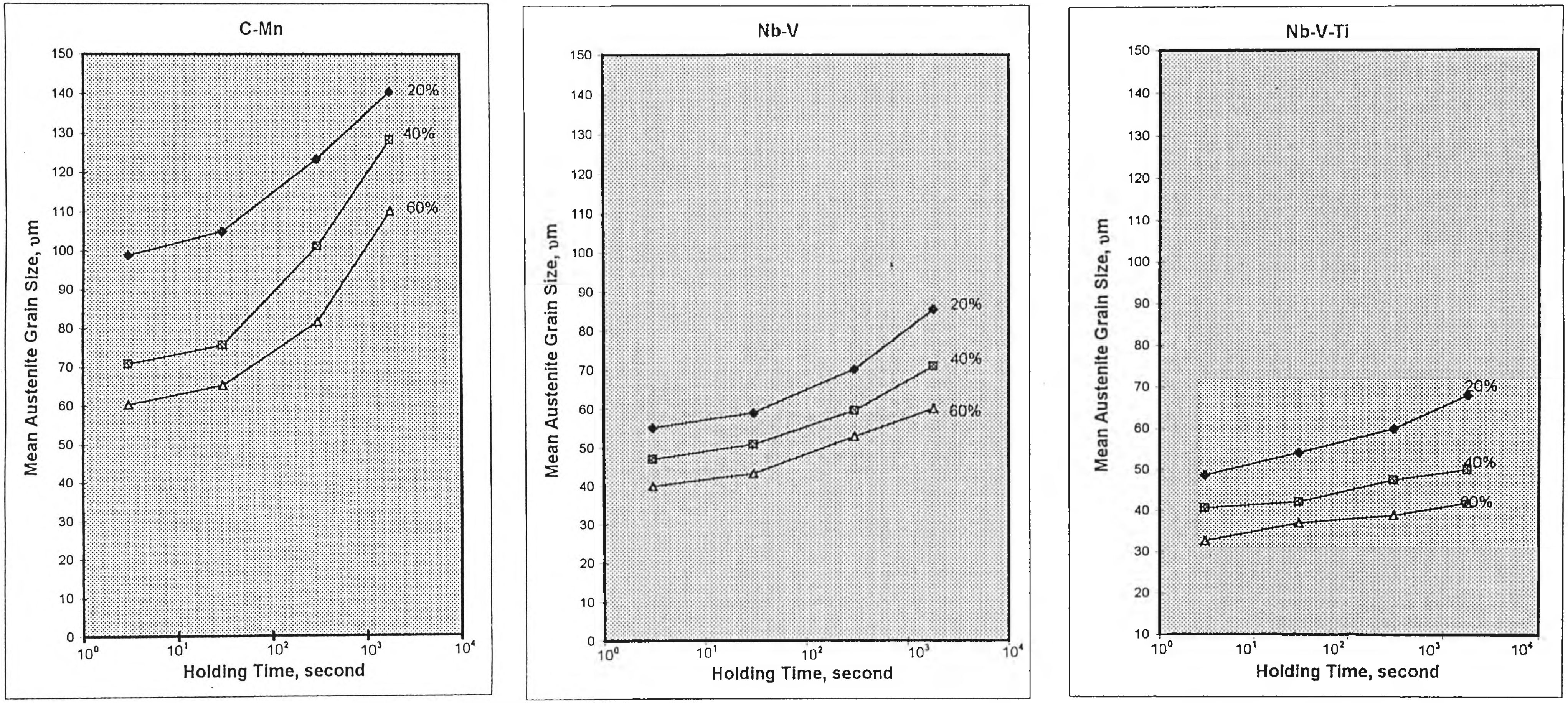

Figure 4.24 Effect of holding time(second) on mean austenite grain size for different reduction, at rolling temperature 1050°C of C-Mn, Nb-V and Nb-V-Ti steel.

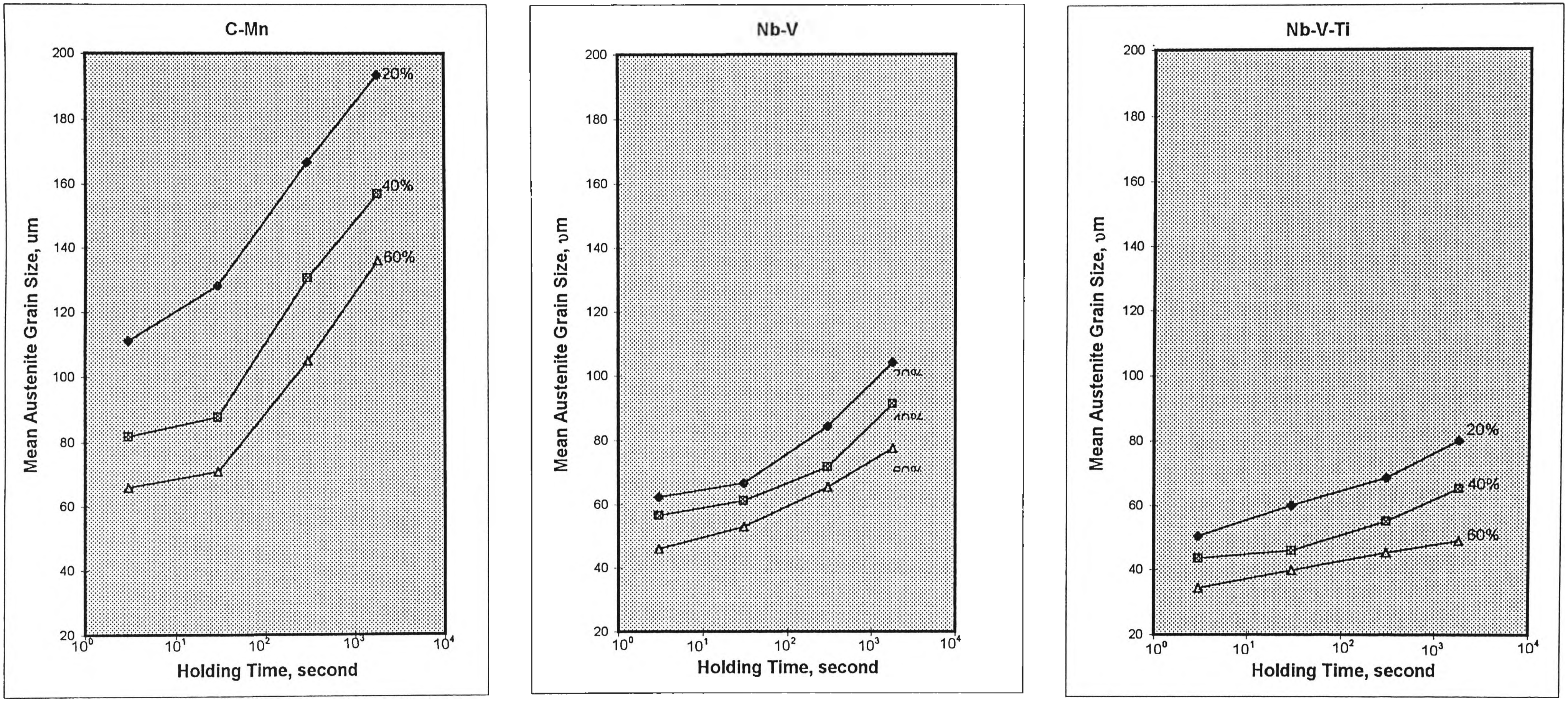

Figure 4.25 Effect of holding time(second) on mean austenite grain size for different reduction, at rolling temperature 1100°C of C-Mn, Nb-V and Nb-V-Ti steel.

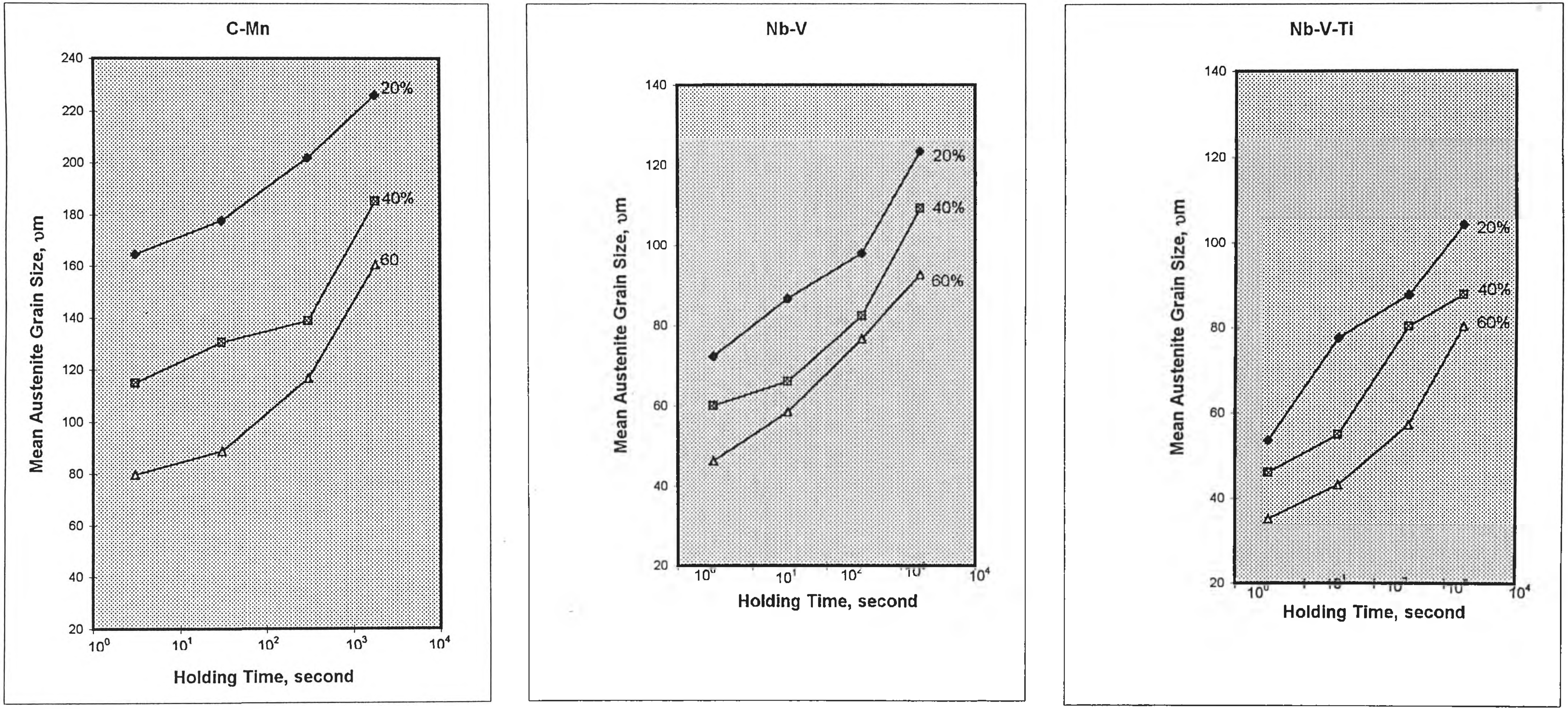

Figure 4.26 Effect of holding time(second) on mean austenite grain size for different reduction, at rolling temperature 1150°C of C-Mn, Nb-V and Nb-V-Ti steel.

CHAPTER 5

DISCUSSION

5. DISCUSSION

5.1. THE EFFECT OF PRECIPITATES ON THE GRAIN COARSENING TEMPERATURE.

The grain growth is a thermally activated process and the grain size which is developed depends both on time and temperature. The grain growth in austenite can be modified considerably by the addition of elements such as alumunium, niobium, vanadium and titanium (22).

The C-Mn steel exibits a relatively low grain coarsening temperature (< 950°C). There seems to be some indication of grain boundary pinning by AlN particles present in the cast steel slab, and the effectiveness of these particles to pin the grain boundary is quite low. And the low stability of AlN at relatively low solution temperature (22), could also influence the precipitation of fine particles during reheating. The effect of the addition of niobium, vanadium and titanium was to raise grain coarsening of austenite, as reported by other workers (5,22,24,28).

One of the reasons for this is that, unlike alumunium, which precipitates only as nitride, niobium, vanadium and titanium form carbides and carbonitrides and the carbon content is generally in excess of stoichiometry (22,23).

In the Nb-V steel which has higher grain coarsening temperature compared to the C-Mn steel it is recognized that niobium carbonitride and vanadium carbonitride particles are carbon-rich (28). Although niobium in solid solution is thought to be able to retard recrystallization, vanadium is a very strong nitrogen fixer, and principally the grain refinement in vanadium steel is due to the presence of vanadium nitride precipitates (23).

The Nb - V steel of this study contained low niobium (Nb = 0.03) and had a low Nb : N ratio of 4.2, which is less than the stoichiometric ratio (6.6). So during solidification of the slab, Nb (CN) is expected to form and all of the niobium would be combined as Nb (CN).

The small vanadium nitrides of the Nb-V steel slab would dissolve first during reheating (up to 1250°C) because the stability of vanadium nitride in austenite is much lower than niobium-carbonitride which would act as a retardator of recrystallization. And finally all of vanadium-nitride and niobium-carbonitride would dissolve in austenite.

The fine vanadium nitride and niobium carbonitride particles dissolve in austenite and thus the effectiveness of grain boundary pinning substantially diminishes. The solution temperature of niobium carbonitride in the Nb - V steel is 1050°C - 1100°C and of vanadium nitride is 950°C - 1000°C (Figure. 2.1), consistent with the grain coarsening temperature of the 0.03 Nb - 0.05 V steel being about 1000°C, (Figure 4.1).

The Nb-V-Ti steel (Nb = 0.05, V = 0.03 and Ti = 0.02) has a grain coarsening temperature slightly higher than that for the Nb - V steel, (Figure 4.1.) and it is recognized that carbonitride particles of niobium, vanadium and titanium are formed. The Nb-V-Ti steel slab had a Ti : N ratio of 2.86 which is less than the stoichiometric ratio (3.42). Therefore, TiN will be formed during solidification of the slab. All titanium will therefore be available to combine with nitrogen to form fine TiN particles. Also, the available niobium combines with excess nitrogen to form Nb (CN), because the affinity of niobium for nitrogen is higher than that of vanadium (15,40,42,43).

During solidification of the Nb-V-Ti steel slab, titanium nitride particles and some niobium carbonitride are expected to form in the high temperature range of austenite. In the lower temperature range of austenite, niobium and vanadium carbide are expected to form, which can be reversed to vanadium nitride, niobium carbonitride and titanium nitride. These nitride and carbonitride particles preferentially nucleate and grow, and at the lower temperature of austenite, nitride and carbonitride particles nucleate more homogeneously.

During reheating (up to 1250°C) precipitates in the Nb-V-Ti steel slab would dissolve in the reverse sequence as that described above. The vanadium nitride particles will dissolve first. Next, small niobium carbonitride particles all dissolve,and with most of niobium carbonitride particles also dissolve small titanium nitride in the austenite, leaving a few coarse titanium nitrides and niobium carbonitrides. Finally, only a few undissolved coarse titanium nitride and niobium carbonitride particles remain resulting in the strongest pining effect.

The grain coarsening temperature starts at the lower limit of solution temperature of titanium nitride and niobium carbonitride. However, the niobium carbonitride particles are less stable than those of titanium nitride. So, undissolved titanium nitride and niobium carbonitride particles can improve strongly the pinning effect at a grain boundary and, together with titanium nitride and niobium carbonitride particles, increase the grain coarsening temperature to a temperature of 1050°C. This temperature is higher than the grain coarsening temperature of Nb-V steel (Figure 4.1).

5.2. AUSTENITE RECRYSTALLIZATION

5.2.1. Effects of Temperature and Deformation

Reheating to 1250°C (which is higher than the grain coarsening temperature of the steels used) resulted in coarser initial austenite grains prior to rolling, and when reheating to a temperature lower than the grain coarsening temperature finer initial austenite grains resulted (Figures 4.12 - 4.14).

The recrystallization behaviour after reheating (1250°C) and single pass rolling is summarized in Figures 4.12 - 4.14. The recrystallization behaviour of C-Mn, Nb - V and Nb-V-Ti steels in as quenched condition is divided into three regions depending on rolling temperature and the amount of reduction, ie. non-recrystallized (NR), partially recrystallized (PR) and recrystallized (R). These regions are separated by recrystallization start (RS) and recrystallization (RF) finish curve (9). The austenite changes from partially to completely recrystallized structure with an increase in reduction or temperature, and critical rolling reduction increases with a decrease of rolling temperature (9).

The recrystallization process is preceded mainly by the motion of dislocation, subgrain, and grain boundaries (23). Therefore, the critical reduction is that when the rolling reduction is higher than critical reduction, the driving force for the motion of dislocations, subgrain and grain boundaries being high enough to overcome barriers from the alloy carbonitride particles (4,7,23).

As can be seen in Figures 4.12 - 4.14, with the expected niobium and titanium precipitates, especially Nb(CN) and TiN particles, in the Nb-V and Nb-V-Ti steels would result in strong subgrain and grain boundary pinning, and critical rolling reduction was higher than in the C - Mn steel. The critical reduction for austenite recrystallization in C - Mn steel is very much lower. The recrystallized austenite grains in the C - Mn steel grew at a higher rate compared with the Nb-V and Nb-V-Ti steels, for the same rolling and holding conditions (Figure 5.1), and this in attributed to the relative absence of strong grain boundary pinning particles.

The critical stop temperature for austenite recrystallization is the temperature below which full recrystallization of deformed austenite will not occur within normal hold times even for rolling with high reductions. For these conditions alloy carbonitride particles provide the main retarding effect on austenite recrystallization (23). In the present work, the critical temperature for austenite recrystallization is taken as a temperature

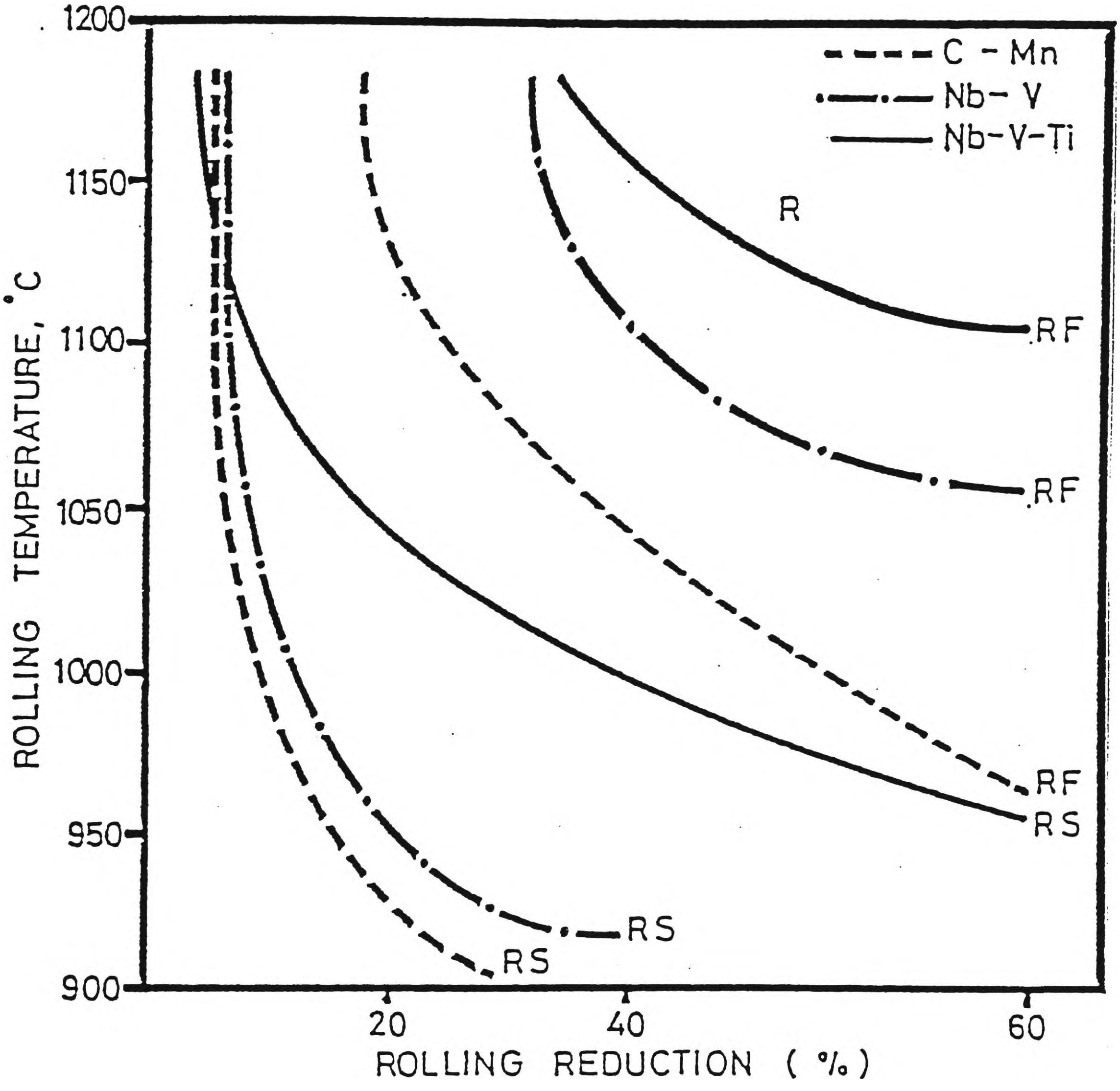

Figure 5.1. **Comparison of Austenite Recrystallization in The Three Steels as a Function of Rolling Temperature and Rolling Reduction. The Samples were Reheated to 1250°C and Rolled with One Pass and Quenched within 3 Sec.**

below which Nb(CN) and TiN precipitation can occur before the onset of austenite recrystallization and exert a strong effect on retarding austenite recrystallization.

The C - Mn steel had very low critical stop temperature for austenite recrystallization, undoubtedly because of the very weak retardation effect of AlN precipitation. The critical temperature for austenite recrystallization of both Nb-V and Nb- V-Ti steel is higher almost certainly due to the stronger pinning effect on austenite grain boundaries of Nb(CN) and TiN particles. The grain growth kinetics of the fully recrystallized austenite grains are more generally described by the Miller equation (24,44).

$$D = K.t^n \qquad (5.1)$$

Where D is the austenite grain diameter (μm), t is holding time (sec) at a given temperature and K and n are constants. The values of n were calculated from the actual data and are given in Figure 5.2, which shows austenite grain size versus holding time.

As the rolling temperature of 950°C was lower than the grain coarsening temperature of the Nb-V and the Nb-V-Ti steels, or lower than the solvus of Nb(CN) and TiN, so in both steels, recrystallized austenite grains grew at a

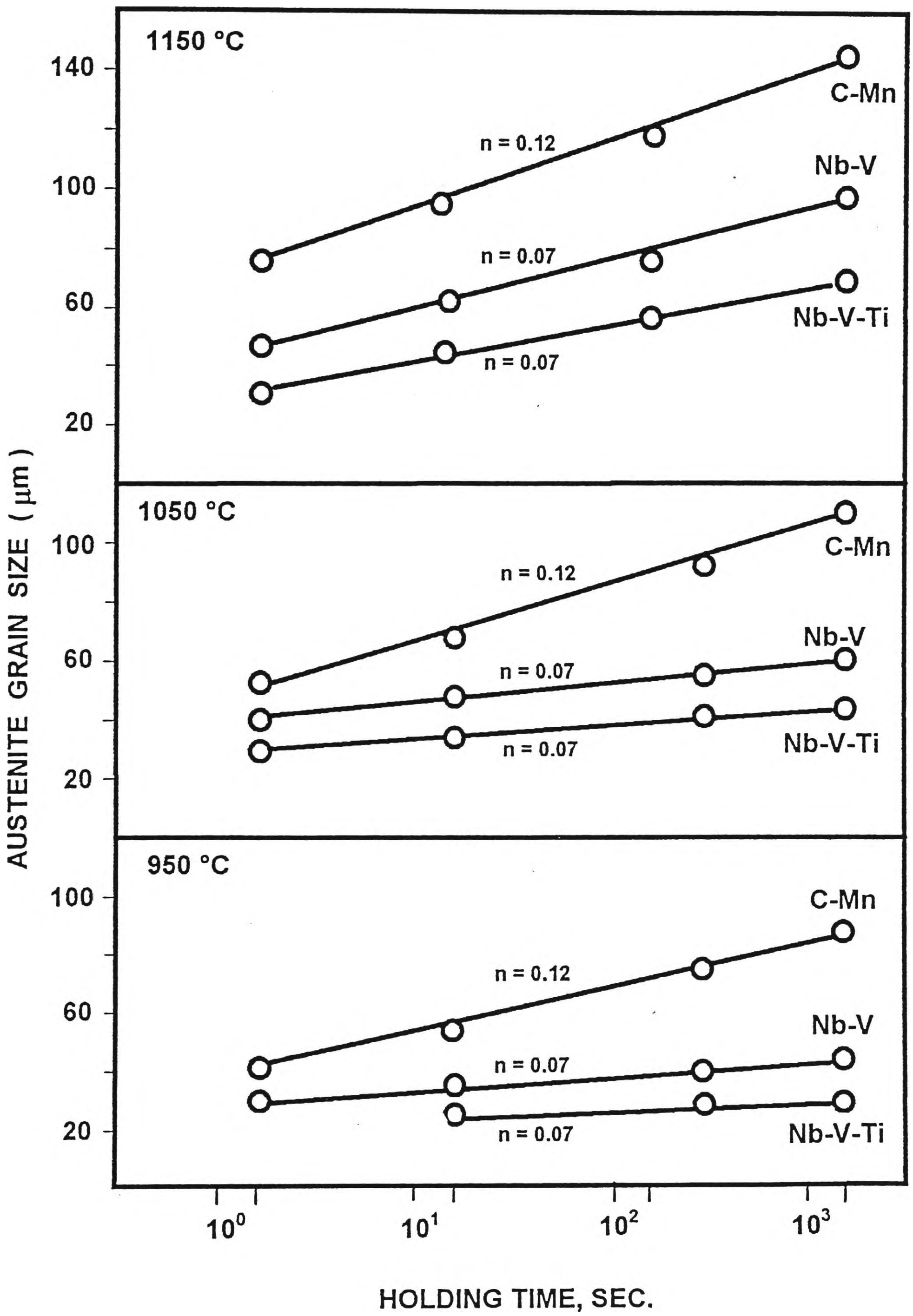

Figure 5.2. Austenite Grain Growth After Recrystallization in C - Mn; Nb - V and Nb - V - Ti Steels, The Reheating Temperature 1250 °C, Rolling 60 % Reduction and Held at The Temperature Indicated.

low rate (n = 0.07) attributed the austenite grain boundary pinning effect of undissolved Nb(CN) and TiN particles.

As the rolling temperature of 1050°C was higher than the solvus for the Nb(CN) and the Nb-V steel so most of the niobium would have been still in solution, and the solute niobium atoms are less effective in retarding the migration of recrystallized austenite. Therefore, the recrystallized austenite grains were associated with a time exponent n = 0.09. As this is higher than in the Nb-V-Ti steel, where the temperature of 1050°C was still, or just below, the solvus of TiN and TiNb(CN), the retardation of migration of recrysallized austenite was expected to be dominant due to pinning effect of undissolved TiN particles, so the recrystaliized austenite grains grew still at a low rate (n = 0.07).

At higher rolling temperature (1150°C) the recrystallized austenite grew with an increased rate to n = 0.12 for the Nb-V steel and n = 0.11 for the Nb-V-Ti steel. The temperature is higher than both of the solvus temperature of both the Nb-V steel and the Nb-V-Ti steel, where mostly the niobium and titanium would be in solution, and less effective in retarding the migration of recrystallized austenite grain boundaries, thus the recrystallized austenite grains grew at higher exponent n (24,44).

5.2.2. Retardation of Recrystallization

The soluted alloying elements produce dislocation drag in the nucleation stage and nitride particles in an alloy result in pinning the nucleation and retard recrystallization (22). The effect of alloying addition was in increasing the critical rolling reduction (Figure 5.1). Because TiN is more stable than NbN and VN (Figure 2.1), so TiN is more effective in increasing the critical rolling reduction in Nb-V-Ti steel than is Nb(CN) in Nb-V steel. The critical rolling reduction also changed with the rolling and holding temperature, and normally decreased with an increase in rolling and holding temperature, as shown in Figure.5.1 and Figure 5.3.

In Figure 4.2, it is shown that after reheating to 1250°C for 15 minutes, rolling at the same reduction and different temperature, and then quenching within 3 sec, the volume fraction of austenite recrystallization was different for the three steels. Therefore, to obtain the same volume fraction of recrystallization, different rolling temperature was needed, the highest temperature for the Nb-V-Ti steel, middle temperature for the Nb-V steel and the lowest for the C-Mn steel.

At rolling temperature of 1050°C, with 20% reduction and quenching within 3 sec, the C - Mn steel was 65% recrystallized, but the Nb-V steel was 25%, and Nb-V-Ti steel was only 10% recrystallized.

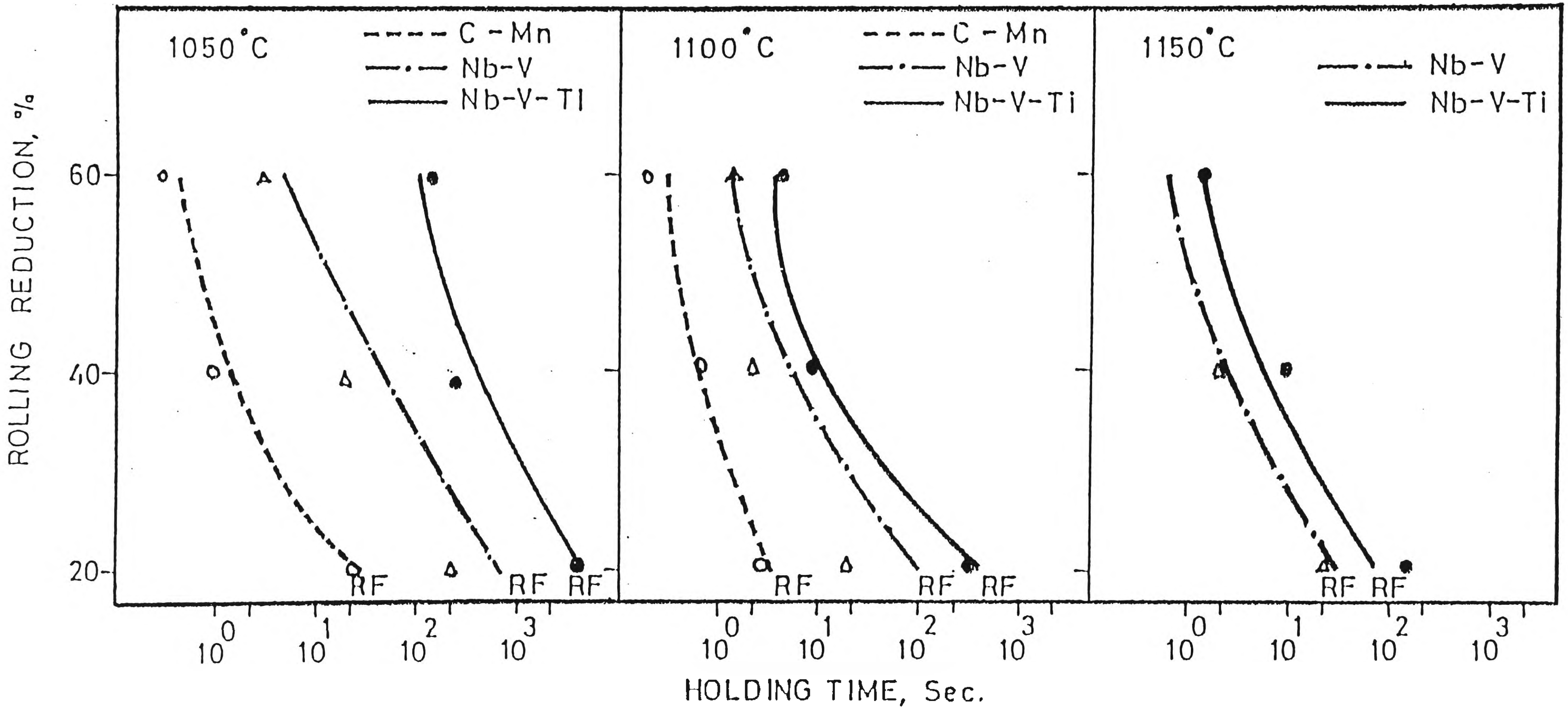

Figure 5.3. Isothermal Recrystallization Kinetics at 1050°C, 1100°C and 1150°C in C-Mn, Nb-V, and Nb-V-Ti Steels Having Various Reductions.

For Nb-V-Ti steel, the amount of reprecipitated and undissolved TiN particles would be quite high, so the effect of grain boundary pinning plays a dominant part in retarding austenite recrystallization.

For a rolling reduction of 20%, the effect of the solution-drag and grain boundary pinning in the Nb-V steel are to be expected to be lower than that of the Nb-V-Ti steel, so retardation of austenite recrystallization in the Nb-V steel is lower. For example, for the same rolling temperature and rolling reduction, the Nb-V steel was 25% recrystallized but Nb-V-Ti steel was only 10% recrystallized.

The higher rolling reduction can substantially refine the austenite grain size. In this case the refinement of austenite grains after heavy rolling would be because of the high nucleation rate associated with the substantial increase of grain surface area per unit volume resulting from the formation of pancake-shaped austenite grains.

When the rolling reduction increased from 20% to 60% at the same rolling temperature of 1050°C (see Figure 4.2), the result was a decrease in the critical rolling reduction where the amount of austenite recrystallized increased as follows : 1) the C - Mn steel was 95% recrystallized, 2) the

Nb-V steel was 85% recrystallized and 3) the Nb-V-Ti steel was 60% recrystallized.

When the rolling temperature for the Nb-V and the Nb-V-Ti steels is lower than the recrystallization temperature, and is therefore performed at the reprecipitation temperatures of Nb(CN) and vanadium nitride or titanium nitride and niobium carbonitride, a strong retarding effect on austenite recrystallization occurs.

At lower rolling temperature (950°C) with higher reduction (60%) and quenching within 3 sec, the C - Mn steel was 90% recrystallized, the Nb-V steel approached 35% recrystallized and the Nb-V-Ti steel was only 8% recrystallized (Figure 4.2).

The time for 95% recrystallization, $t_{0.95}$, was calculated for the C-Mn steel by using the empirical equation proposed by Sellars (9).

$$t_{0.95} = 5.2 \times 10^{-19} d_0^2 \in^{-4} \exp 300.000/RT. \qquad (5.2)$$

Where d_0 is the austenite grain size prior to deformation to an equivalent true strain at temperature T (K), and the activation energy Q-rex = 300.000 J/mole. The calculated values are shown as open circles in Figure 5.4.

The curves of the kinetics of recrystallization (RF) for different steel at various temperatures and quenching within 3 sec. are as shown in Figure 5.5.

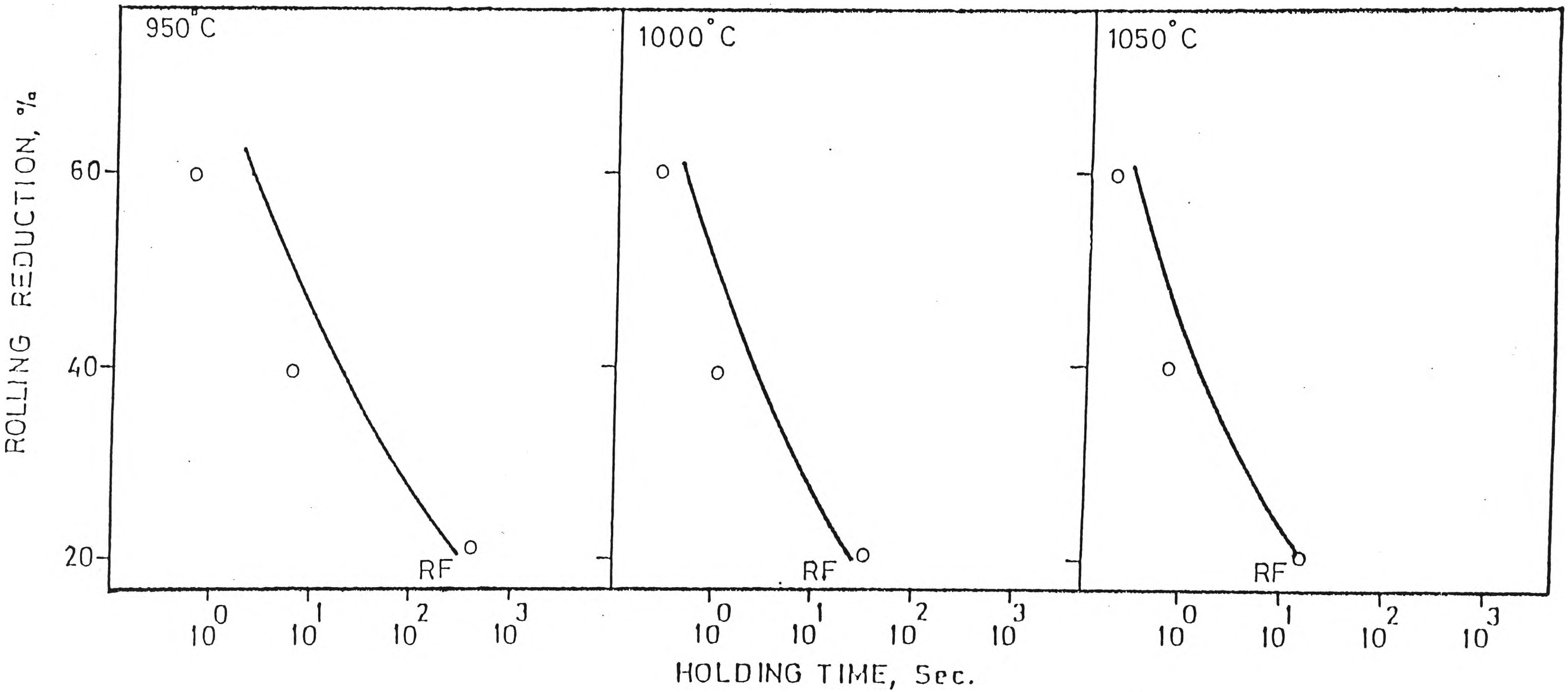

Figure 5.4. Isothermal Recrystallization Kinetics at 950°C, 1000°C and 1050°C in C-Mn Steel Having Various Reductions. [Circle Show Theoretical Prediction for 95% 'Recrystallization in C-Mn Steel Based on Sellars Equation]

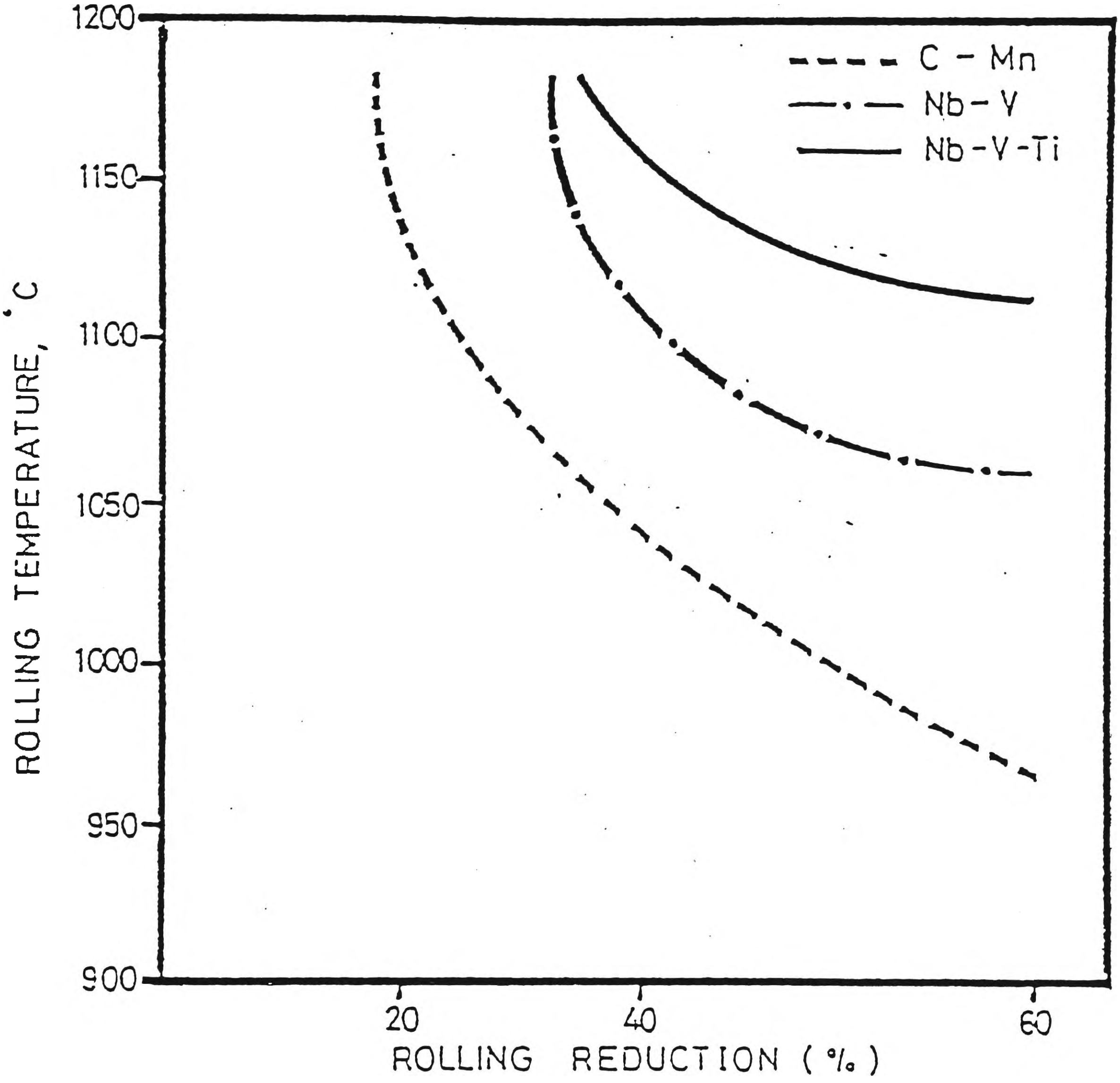

Figure 5.5. Plot of Rolling Temperature versus Rolling Reduction on The Critical Reduction for Recrystallization (RF) for The Different Steels.

5.3. THE MEAN AUSTENITE GRAIN SIZE

5.3.1. Effect of Rolling Temperature on The Mean Austenite Grain Size.

The austenite recrystallization is a result of hot rolling and depends on the material and the rolling variables. The recrystallization may be complete, partial or recrystallization may not occur. The austenite grain size can change significantly as the volume fraction of recrystallized grains increases and as grain growth occurs on holding after hot rolling. After reheating at high temperature, and cooling to lower temperature of rolling (950°C), the austenite in the non-recrystallized state, where the original grains are elongated in the rolling direction and the mean grain size approximates the initial size. (23,45).

At the higher rolling temperature (≥ 1000°C), recrystallization starts readily and continues until completion. If the volume fraction of recrystallized austenite increases, the grain size decreases continuously until complete recrystallization has occurred, and this behaviour depends on the amount of deformation. At low temperature, a high deformation causes rapid decrease in grain size, whereas low deformation requires a high temperature (45). The mean recrystallized austenite grain size for the C-Mn, Nb-V, and Nb-V-Ti steels increases with increasing temperature of deformation (Figure 4.15). The C-Mn curve is steeper than the Nb-V and the Nb-V-Ti steel, and the curve of the Nb-V steel is higher than the Nb-V-Ti steel. Also Figure 4.16 to Figure 4.18 show the effect of rolling temperature of samples quenched within 30, 300 and 1800 sec. From these figures it is clear that the mean recrystallized austenite grain size increases with an increasing temperature of deformation and holding time.

The mean austenite grain size vs rolling temperature curves obtained on direct quenching after 20%, 40% and 60% reduction for C-Mn, Nb-V and Nb-V- Ti steels are shown in Figure 4.15. When the temperature was increased from 950°C up to 1150°C, the normal grain growth was present in the C-Mn steel. The recrystallized grain size was essentially unaffected by rolling temperature in the Nb-V steel and the Nb-V-Ti steel for a rolling temperature in the range from 950°C up to 1050°C, but when rolling temperature increased to 1150°C, some grain growth was evident. As expected, the recrystallized grains were finer in the three steels in 60% deformation samples compared to those with 20% and 40% reduction. And there were similar trends in the three steels when the holding time increased after rolling up to 1800 sec.

The Nb-V steel and the Nb-V-Ti steel exibited only a slight increase in recrystallized grain size and this can be explained by nothing that the samples were transferred to the bath were in the direct quenched condition or quenched within 3 sec. delay after rolling. Although in this case the precipitation of the carbonitride particles would not have had time to occur (41), the grain pinning effect is probably due to undissolved nitride particles, such as Nb(CN) and TiN particles which when present pin austenite grain boundary and retard grain growth (23,44).

When the holding time increased from 30 up to 1800 sec. a similar trend is shown in the C-Mn, the Nb-V and the Nb-V- Ti steels, attributed to increasing grain growth (Figure 4.22 up to Figure 4.26).

The grain growth after full recrystallization was negligible in the Nb-V steel and the Nb-V-Ti steel at temperatures below 1050°C (Figures 4.22-4.23) In the C-Mn steel, the recrystallization process is very fast and even the quench time of 3 sec provided sufficient time for extensive grain growth (8,20,32).

The formation of mixed austenite grain structure and partial recrystallization of austenite is caused by strain induced grain boundary migration during holding (46,47). The mixed grain structure exists in a wider range for the Nb-V and the Nb-V-Ti steels when the temperature is over the range of reduction, because process of recrystallization in the Nb-V and the Nb-V-Ti steels is very sluggish so that partial recrystallization extends that range of conditions more than for the C-Mn steel. When an actual reduction is higher than the amount of reduction required to start recrystallization,

recrystallization occurs at various selective locations, and this produces a mixed grain structure by fine recrystallized grains and unrecrystallized grains (23,46). This effect can be seen in Figure 4.7.

5.3.2. Effect of Rolling Reduction

The effect of amount of reduction by single pass rolling at various temperatures on mean austenite grain size is shown in Figures 4.19-4.21 with different holding time after rolling. These results show that the recrystallized grain size decreases continuously with increase in the amount of reduction in all the three steel.

The austenite grain surface area per unit volume, Sgb, can be expressed by austenite grain diameter D as (24,48,49) :

$$S_{g.b} = 4 / (\sqrt{\pi} . D) \tag{5.3}$$

The grain boundary surface area of austenite per unit volume of a specimen increases on deformation. When the surface area is plotted as a function of rolling reduction in Figure 5.6, it is seen that the grain surface area per unit volume increases with the increase in rolling reduction, and the increase is higher when the reduction is higher and equation 5.3 is only valid for equiaxed (recrystallized) grains, for elongated grains with the same average diameter D, $S_{g.b}$ will be higher and are different formula are required (24).

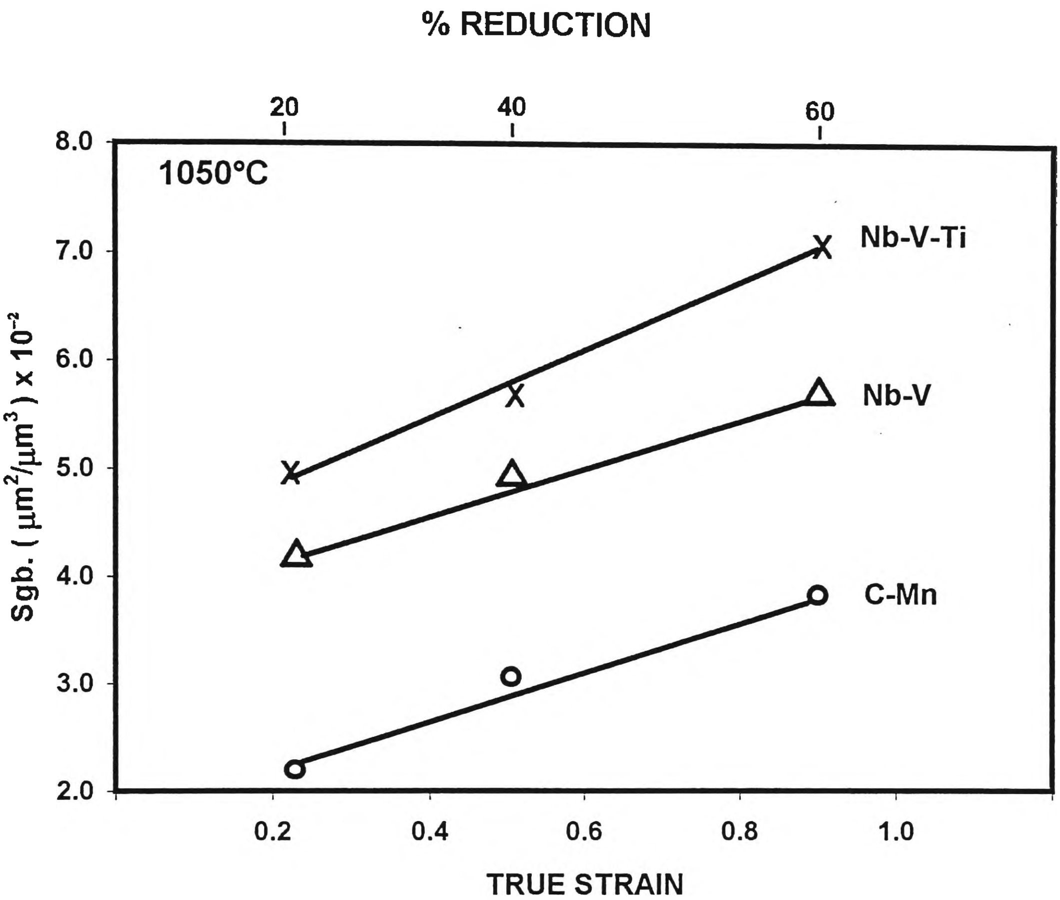

Figure 5.6. The Austenite Grain Surface Area per Unit Volume as a Function of Rolling Reduction for C-Mn, Nb-V and Nb-V-Ti Steels at 1050°C.

The higher rolling reduction can produce finer austenite grain size. The refinement of austenite grain size in the present samples was caused by the high nucleation rate and associated with the substantial increase of grain surface area per unit volume resulting from the formation of pancake-shaped austenite grains (24).

In Figure 4.19 the recrystallized grain size curves are not as steep in the range of lower rolling temperature 950°C and 1050°C for the Nb-V and the Nb-V-Ti steels compared with curves at higher temperature of rolling. When the holding time after rolling increased up to 300 sec. a similar trend is shown in the three steels.

When rolling at a lower temperature than the recrystallization temperature, the result is a strong retarding effect on austenite recrystallization by reprecipitation of nitride and carbonitride in the Nb-V and the Nb-V-Ti steels (23,24).

5.3.3. Effect of Holding Time

The effect of holding time at the rolling temperature after deformation on mean austenite grain size is shown in Figures 4.22 to 4.26 . At the rolling temperature of 950°C it is shown in Figure 4.22 that the slope of curves of both the Nb-V and the Nb-V- Ti steels for 40% and 60% rolling reduction is, on average, lower than the slope of curves at present higher rolling temperature at the same rolling reduction with holding time up to 1800 sec. (Figure 4.25).

In the Nb-V-Ti steel, increase in the slope of curves for 40% and 60% rolling reduction is small for an increase of rolling temperature from 950°C up to 1050°C, for holding times up to 1800 sec. (Figures 4.22 - 4.24)

When the holding time is increased in the non-recrystallization rolling condition, where the steel starts recrystallization, the resulting mean austenite grain size decreases continuously until the critical time for complete recrystallization (9). In the C-Mn steel, the rate of decrease in grain size with time was generally lower than for the Nb-V and the Nb-V-Ti steel because of grain growth occurring simultaneously with recrystallization, whereas the C-Mn steel slope of curves is higher than that of the other to steels (Figures 4.22 - 4.24). When the complete recrystallization is reached, with continuously holding the sample at the rolling temperature produces the grain growth.

The mean recrystallized grain size remained unaffected with holding time for the Nb-V and the Nb-V-Ti steels rolled at temperature below the solvus temperature, and only the small post-recrystallization grain growth occured in 40% and 60% rolled samples at 1000°C for the Nb-V steel, and at 1050°C and below for the Nb-V-Ti steel.

5.4. THE ACTIVATION ENERGY FOR 50% RECRYSTALLIZATION OF AUSTENITE

Time for 50% volume fraction recrystallization ($t_{0.50}$) for the C-Mn, the Nb-V and the Nb-V-Ti steels were estimated from Figures 4.2 - 4.5, and are shown in Figures 5.7,5.8 and 5.9. It can be seen that the expected change of slope in the Nb-V steel (9) coresponds to a temperature of approximately 990°C, and for the Nb-V-Ti steel to that of approximately 1030°C, and reduction in the range of 40% - 60% (Figures 5.8 and 5.9). The grain coarsening temperature with out reduction of the Nb-V steel is 1000°C and the Nb-V-Ti steel is 1050°C (from Figures 4.1).

The retardation of recrystallization below about 990°C in the Nb-V steel and 1030°C in the Nb-V-Ti steel greatly increases for both steels. (Figures 5.8 and 5.9). This type of behaviour has been interpreted in terms of the onset of strain induced precipitation which interacts with, and pins the deformation structure, retarding the recrystallization process (9). It is also consistent with the analysis by Sellars and Beynon of static recrystallization in titanium steels under hot working conditions (50).

This temperature is interpreted as that below which the volume fraction of fine precipitates is sufficiently high to stabilise the deformed austenite structure with respect to static recrystallization .

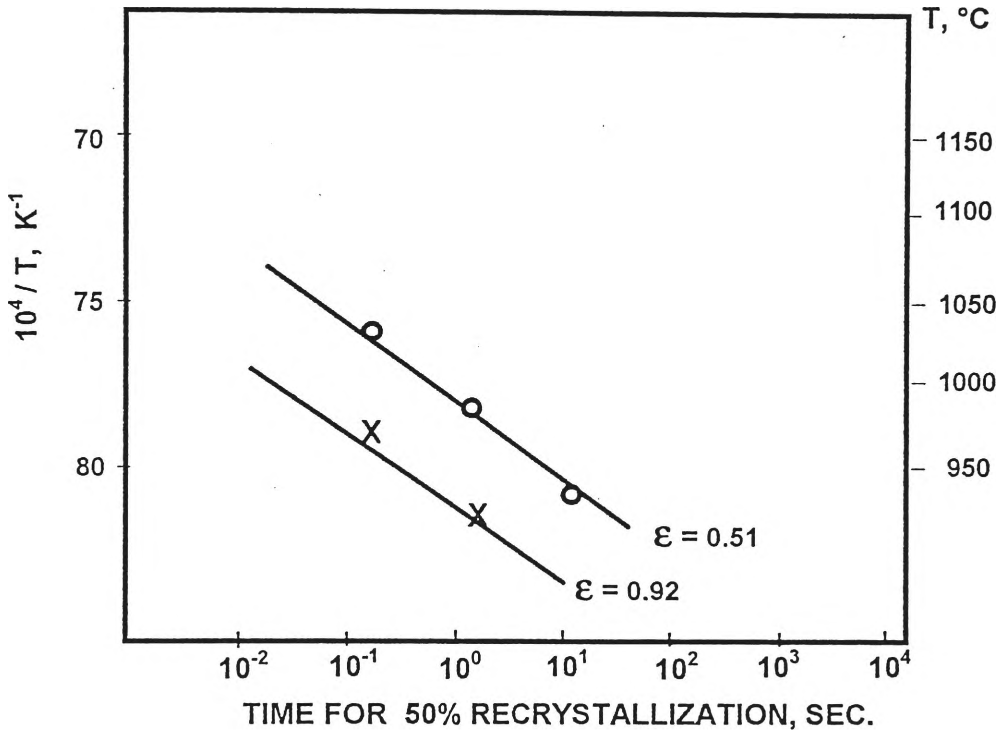

Figure 5.7. Temperature Dependence of Time to 50 % Recrystallization in The C - Mn Steel at 60%, 40% and 20 % Reduction.

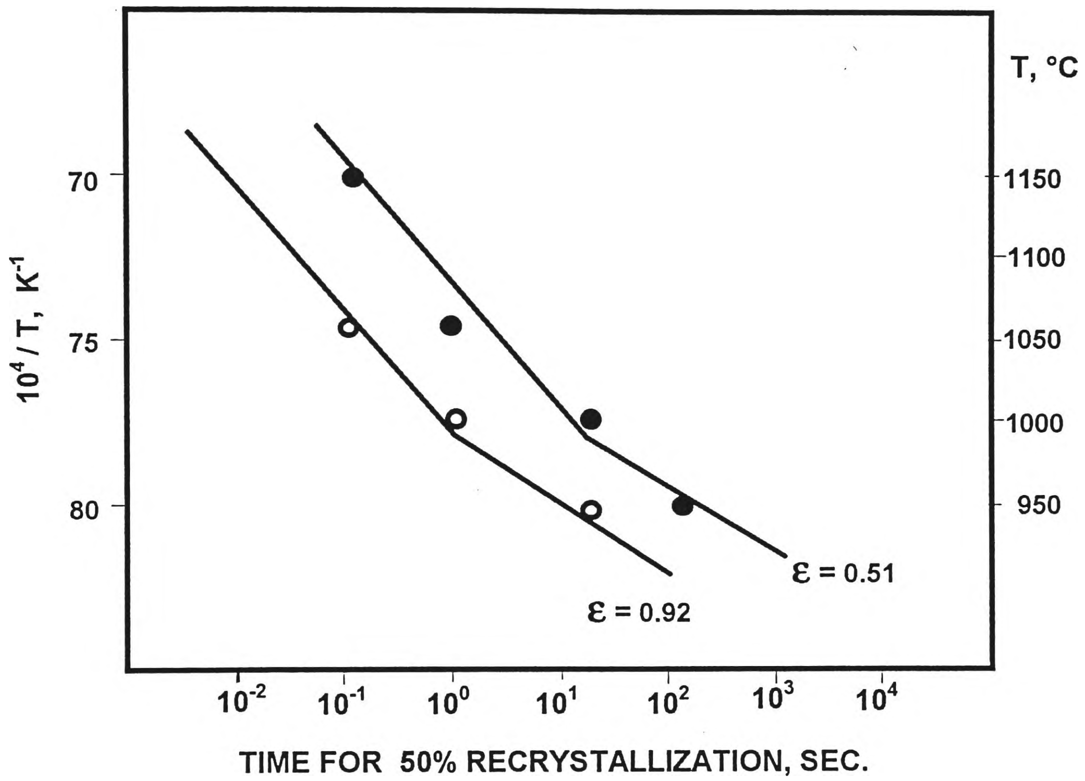

Figure 5.8. Temperature Dependence of Time to 50 % Recrystallization in The Nb - V Steel at 60% and 40% Reduction.

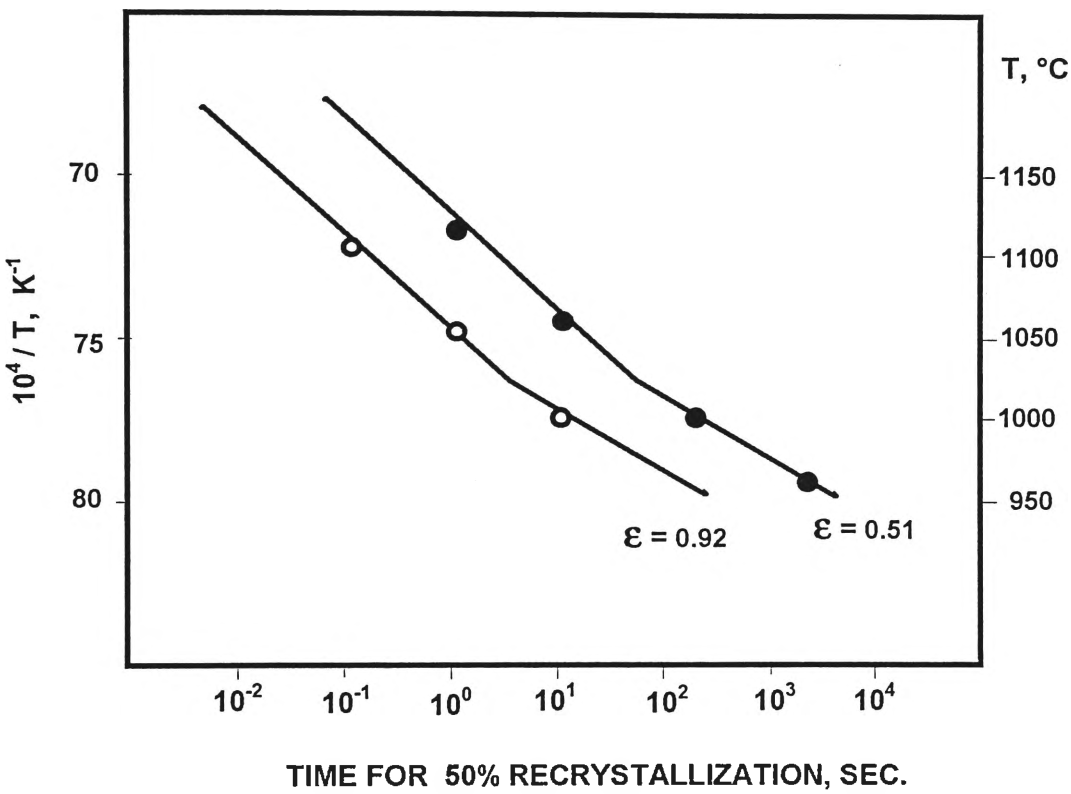

Figure 5.9. Temperature Dependence of Time to 50 % Recrystallization in The Nb - V - Ti Steel at 60% and 40 % Reduction.

The slope of the lines in Figures 5.8 and 5.9 were used to calculate the activation energy for 50% recrystallization of austenite according to equation 5.4 (51).

$$t_{0.5} = \alpha \, . \, e^{Q/RT} \qquad (5.4)$$

Where : $t_{0.5}$ = time for 50% recrystallization

Q = activation energy

R = 8.2063 J x mole^{-1} x K^{-1}

T = rolling and holding temperature (K)

The calculated Q-rex (activation energy) of the C-Mn steel was approximately constant at about 300 KJ/mole and consistent with other measurements (45,51). See Figure 5.7. The activation energy showed some strain or reduction dependence. At higher strains, the energy barrier opposing recrystallization is smaller, but still large for steels in which precipitation does not interact with recrystallization. (5.4). From the slope of curves in Figure 5.8 and Figure 5.9, the activation energies for 50% recrystallization (Q-rex) of the Nb-V and the Nb-V-Ti steels were calculated according to equation (5.4), and listed in Table 5.1.

Table. 5.1. Activation Energy for 50% Recrystallization, Q-Rex (KJ/mole) of Nb-V and Nb-V-Ti Steels.

Steel	Tp	Q-rex ($t > T_p$)		Q-rex ($t < T_p$)	
		$\varepsilon = 0.92$	$\varepsilon = 0.51$	$\varepsilon = 0.92$	$\varepsilon = 0.51$
Nb - V	990°C	320	560	410	710
Nb-V-Ti	1030°C	340	620	470	855

For the Nb-V steel with reduction 60%, the average value of Q-rex, was 320 KJ/mole above 990°C and was 410 KJ/mole below 990°C. For the Nb-V-Ti steel, the average value of Q-rex, was 340 KJ/mole above 1030°C and was 470 KJ/mole below 1030°C with same reduction.

The higher activation energy at low reduction implies that precipitate particles are very effective in stabilizing the deformation structure of austenite in which the driving force for recrystallization, in the from of stored strain energy, is relatively low.

The high activation energies (Q-rex) at strain $\in$ = 0.51 are about 2 times higher than at the high strain ($\in$ = 0.92) at same rolling temperature. The strain dependence of Q - rex measured, decreased sharply from 620 KJ/mole at 40% reduction ($\in$ = 0.51) to 340 KJ/mol at 60% reduction ($\in$ = 0.92). With temperature above temperature strain induced precipitation (T_p) of 1030°C Q - rex decreased sharply from 560 KJ/mol to 320 KJ/mole in the Nb-V steel, where temperature was above 990°C.

Strain dependence of the activation of nucleation and growth of recrystallized grains, is such that activation energies are increased as strain is decreased and the activation energy for nucleation of recrystallization is more strongly strain dependent than it is for growth. (51).

5.5. THE PREDICTION OF TIME FOR 50% RECRYSTALLIZATION

Static recrystallization kinetics depend strongly on strain (driving force) and on pre-existing grain size and temperature. Sellars (24) has given relation of recrystallization kinetics ($t_{0.5}$) in niobium steel for 50% recrystallization as:

$$t_{0.5} = 2.52 \times 10^{-19}\, d_0^{2}\, \epsilon^{-4} \exp 325000/RT \qquad (5.5)$$

Where : d_0 is initial diameter grain size (μm)

ϵ is true strain

For the titanium steel, Roberts et al (50) give the relationship

$$t_{0.5} = 5 \times 10^{-18}\, d_0^{2} (\epsilon - 0.058)^{-3.5} \exp 280000/RT \qquad (5.6)$$

The experimental values of Q-rex of the present investigation given in Section 4.3, at low strain (ϵ = 0.51) was about 2 times higher than the value at high strain (ϵ = 0.92).

Assuming that the value of Q-rex used at high strain can also be used at low strain in the the equations (5. 5) and (5.6) i.e. 320 KJ/mole for the Nb-V steel and 340 KJ/mole the NbVTi steel, then the calculated times for predicting the 50% recrystallization time are as listed in Table 5.2.

Table. 5.2. Predicted Values of Time to 50% Recrystallization C-Mn, Nb-V and Nb-V-Ti Steels.

Temp. (°C)	Strain ($\in$)	$t_{0.5}$ (sec.)		
		C-Mn	Nb-V	Nb-V-Ti
950	0.92	0.28	0.71	112
	0.51	2.97	10.54	1060
	0.22	82	408	58600
1000	0.92	0.08	0.28	30
	0.51	0.92	3.15	678
	0.22	25.3	58.5	10100
1050	0.92	0.03	0.07	7.1
	0.51	0.31	0.90	80
	0.22	8.5	18.5	2000
1100	0.92	0.01	0.02	0.95
	0.51	0.11	0.40	20.5
	0.22	3.10	6.20	380
1150	0.92	0.0042	0.007	0.28
	0.51	0.05	0.10	7.0
	0.22	1.2	1.3	130

The prediction of equations (5, 5) and (5.6) for the Nb-V steel and the Nb-V-Ti steel on strain dependence of time to 50% recrystallization and a comparison of the measured values is shown in Figures 5.10 and 5.11.

Since the predicted times for 50% recrystallization for the Nb-V steel for various reductions by Sellars equation (see Figure 5.10) were very close to measured values for temperature $\geq$ 1000°C, it can therefore be seen that the Sellars equation is in good egreement with the measured values.

In Figure 5.11 the predicted times for 50% recrystallization for the Nb-V-Ti, steel for various reductions were slightly higher than the measured values. Equation (5.6) provided by Roberts et.al (50), gave the similar prediction of the time for 50% recrystallization with observed values for the Nb-V-Ti steels. The predicted recrystallization times to 50% recrystallized differ by about 100 times between the Nb-V steel and the Nb-V-Ti steel. On the other hand, the measured values of recrystallization times differ by about 10 times higher. It may be that for closer prediction the constant of equation (5.6.) may have to be modified.

The time for 50% recrystallization of C-Mn steel is described by Sellars (9,24) as :

$$t_{0.5} = 2.5 \times 10^{-19} d_0^2 \in^{-4} \exp 300000/RT \qquad (5.7)$$

It must be remembered that in equation (5.7) the activation energy for recrystallization of 300 KJ/mole is a mean value obtained from the available data of temperature dependency of time for 50% recrystallization.

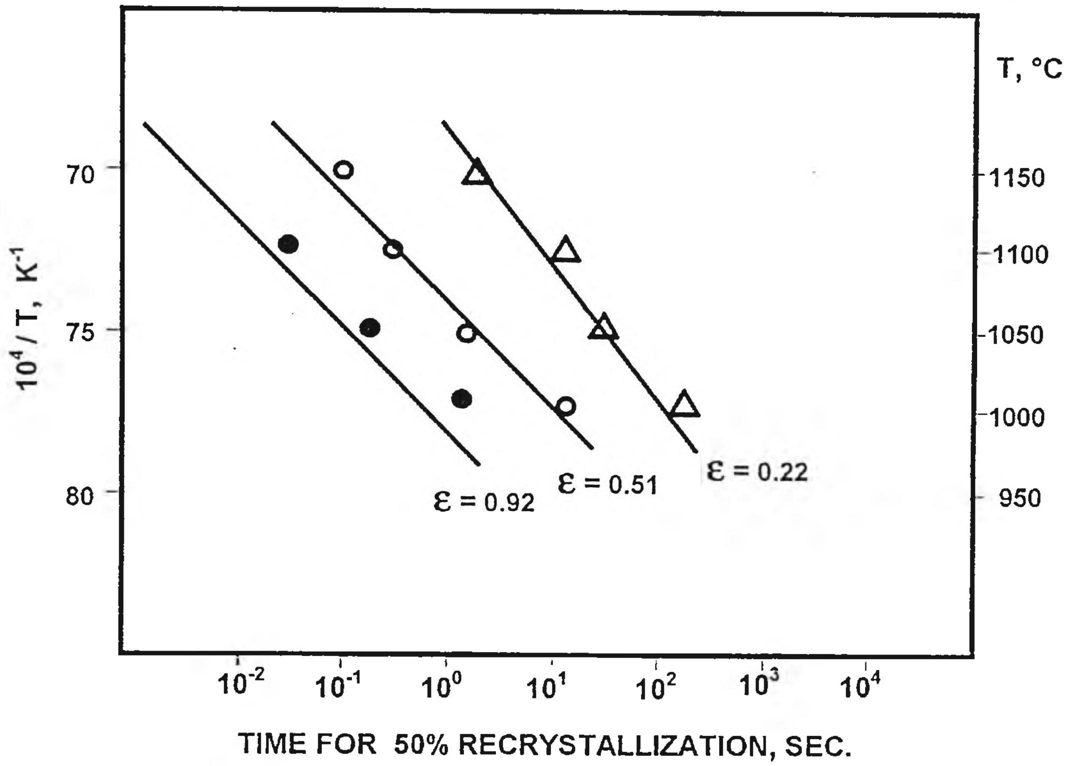

Figure 5.10. Predicted and Measured Times For 50% Recrystallization for the Nb - V Steel For 60 %, 40 % and 20 % Reduction. Symbols Show Measured Values.

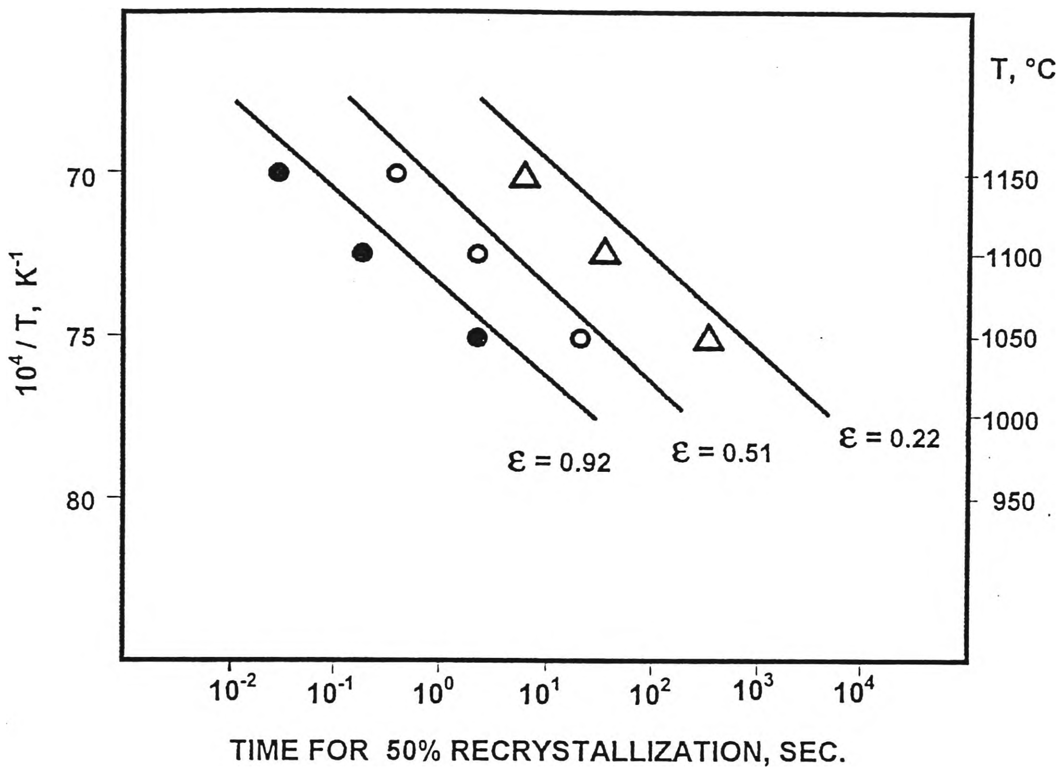

Figure 5.11. Predicted and Measured Times For 50% Recrystallization for The Nb - V - Ti Steel For 60%, 40 % and 20 % Reduction. Symbols Show Measured Values.

The prediction of equation (5.7) for the C-Mn steel on strain and rolling temperature dependency of time for 50% recrystallization are also listed in Table 5.2.

Figure 5.12 shows the time for 50% recrystallization for the C-Mn steel plotted against strain, and it is evident that $t_{0.5}$ is sensitive to strain ($\in$) and at 950°C it appears slightly more so. The mechanism for the retardation of recrystallization in the C-Mn steel is similar to that occurring in the Nb-V and the Nb-V-ti steels, and, as noted earlies, is due to the influence of a precipitation process, where AlN precipitation is responsible for the recrystallization kinetics at rolling temperature below 950°C.

Table 5.2, shows comparison of the behaviour of time for 50% recrystallization of the Nb-V and the Nb-V-Ti steels which is similar to the behaviour of the C-Mn steel with respect to strain dependency, exhibited in Figures 5.13 and 5.14, and obtained by Sellars et.al. equations. The time for 50% recrystallization is seen to decrease by increasing the value of true strain at same temperature for both steels, (the Nb-V and the Nb-V-Ti steels).

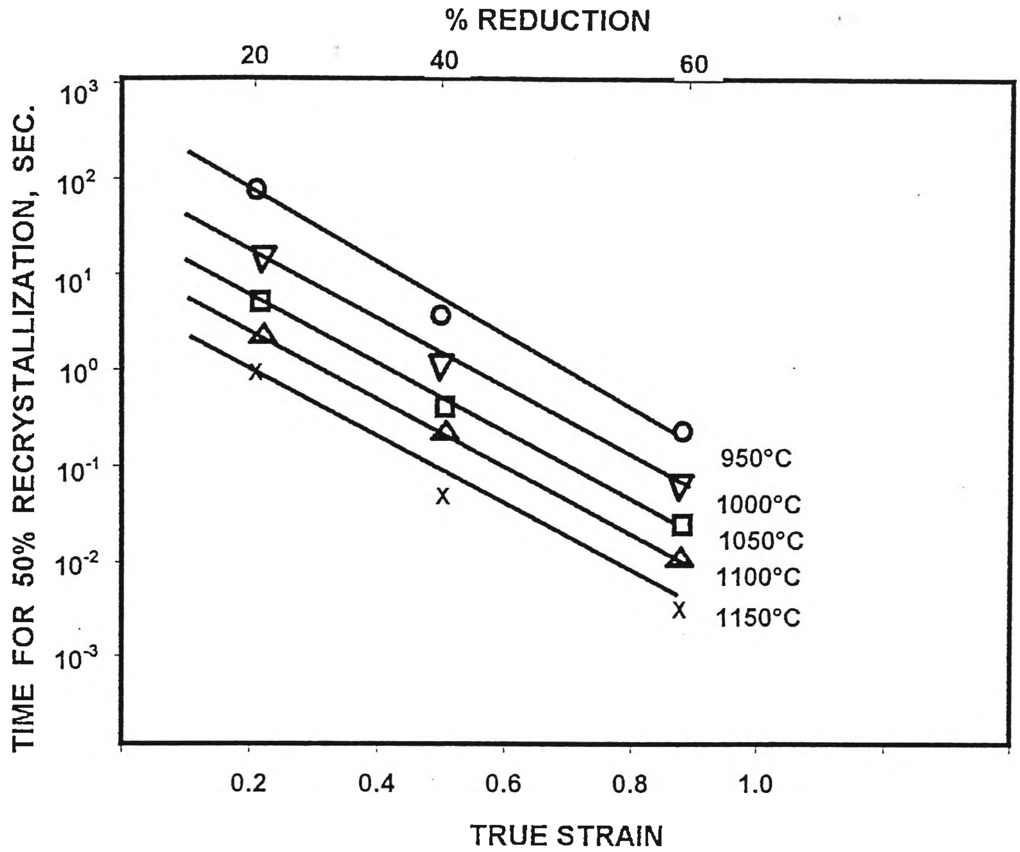

Figure 5.12. Strain Dependence of Time to 50% Recrystallization in The C - Mn Steel (Full lines are predicted by Sellars's equation).

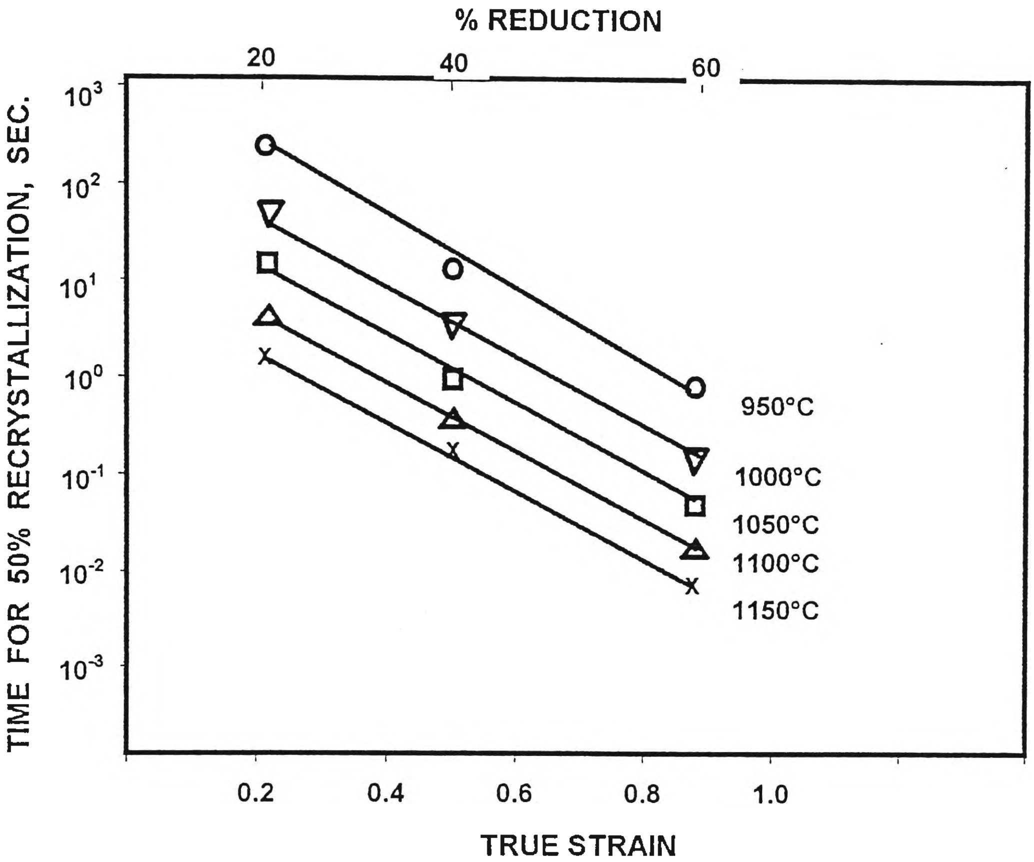

Figure 5.13. Strain Dependence of Time to 50% Recrystallization in The Nb - V Steel (Full lines are predicted by Sellars's equation).

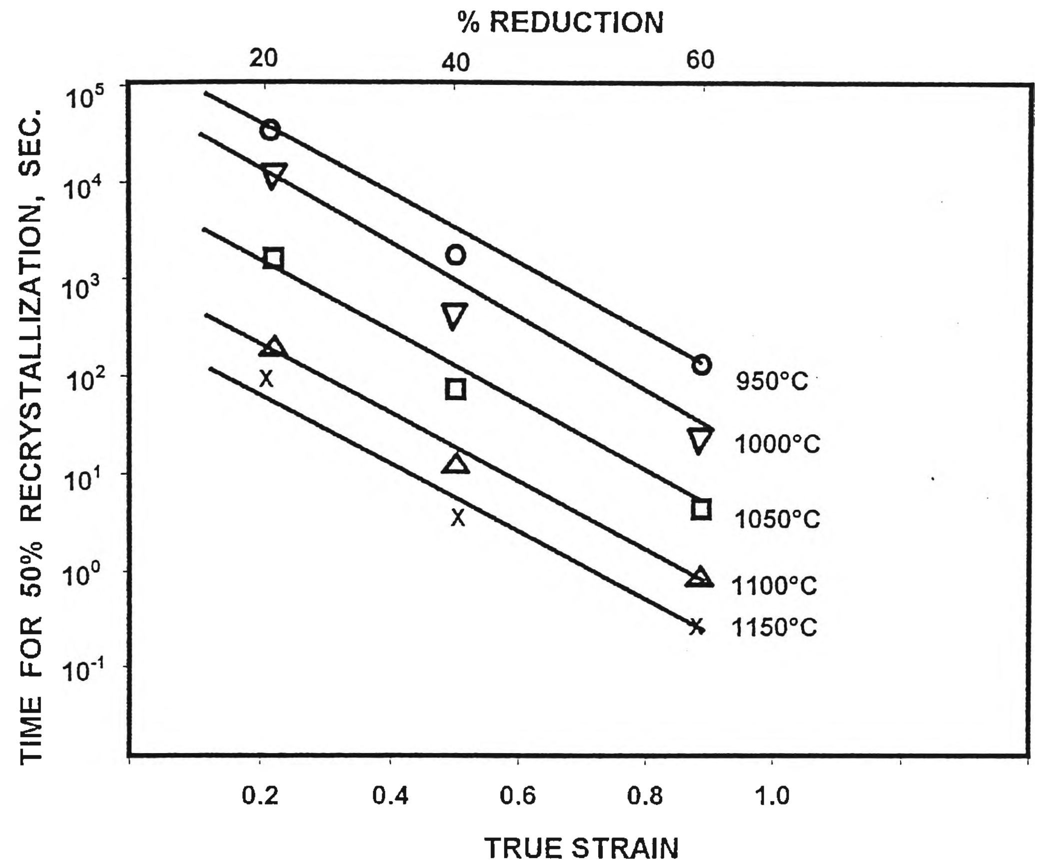

Figure 5.14. Strain Dependence of Time to 50% Recrystallization in The Nb - V Steel (Full lines are predicted by Siwecki's equation).

The kinetics of static recrystallization in hot working can be well described by the Avrami equation, where the dependency of recrystallized fraction on the time (t) and ($t_{0.5}$) during recrystallization (52,53), is given by :

$$X = 1 - \exp\left[-0.693(t/t_{0.5})^{n} \right] \tag{5.8}$$

Where X is the volume fraction recrystallized after time t, whereas the time for 50 % recrystallized is $t_{0.5}$, and the Avrami exponent, n, may be regarded as a material parameter. Values for n reported in the literature lie between 1.5 and 2.0 for C-Mn and microalloyed constructional steels, and for the present purposes an average value of 1.7 is considered appropriate and in line with lieterature (52).

Figure 5.15 shows the influence of temperature on th static recrystallization kinetics for fraction recrystallized following Avrami's equation for Nb-V steels, true strain 0.5 and time for 50% recrystallization ($t_{0.5}$) adopted from Table 5.2.

The influence of temperature on the static recrystallization kinetics for fraction recrystallized following the Avrami equation for Nb-V-Ti steel with true strain 0.51 is shown in Figure 5.16.

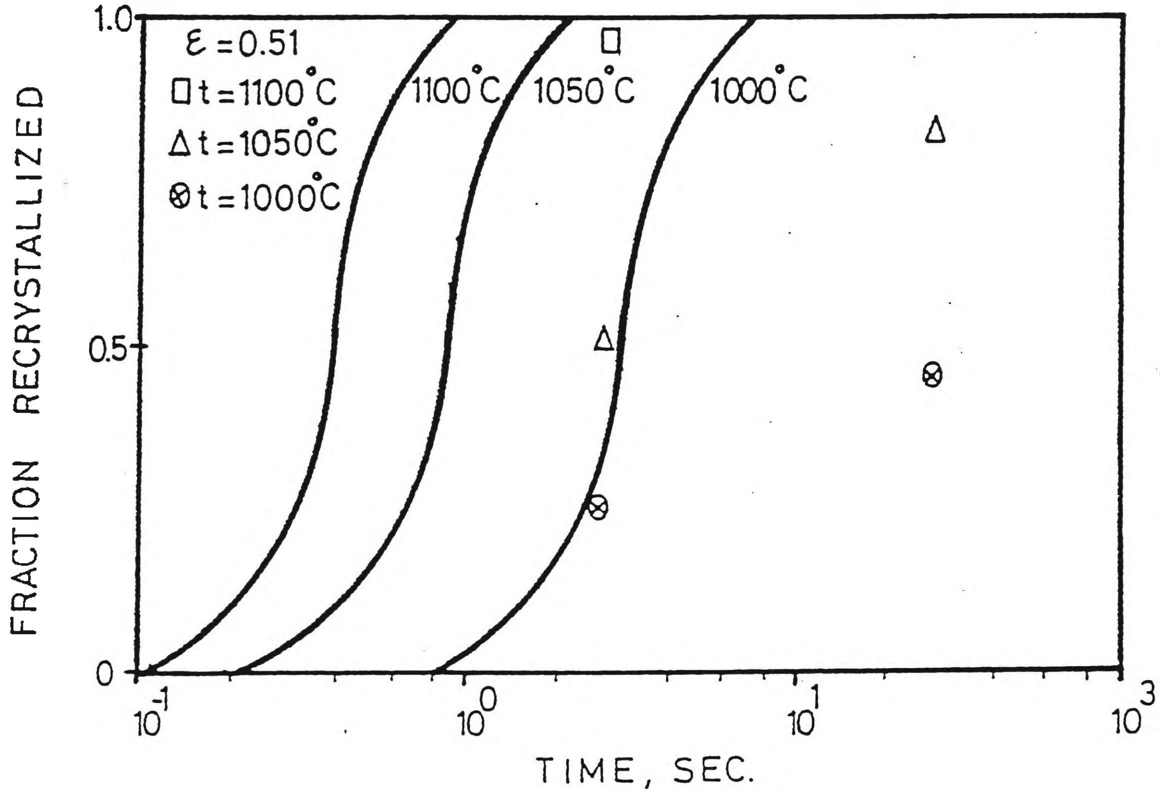

Figure 5.15. Kinetics of Static Recrystallization Following an AVRAMI Relationship [Eq.5.8] for Nb-V Steel at 1000 °C, 1050 °C and 1100 °C of Strain 0.51 Symbols Show Measured Values.

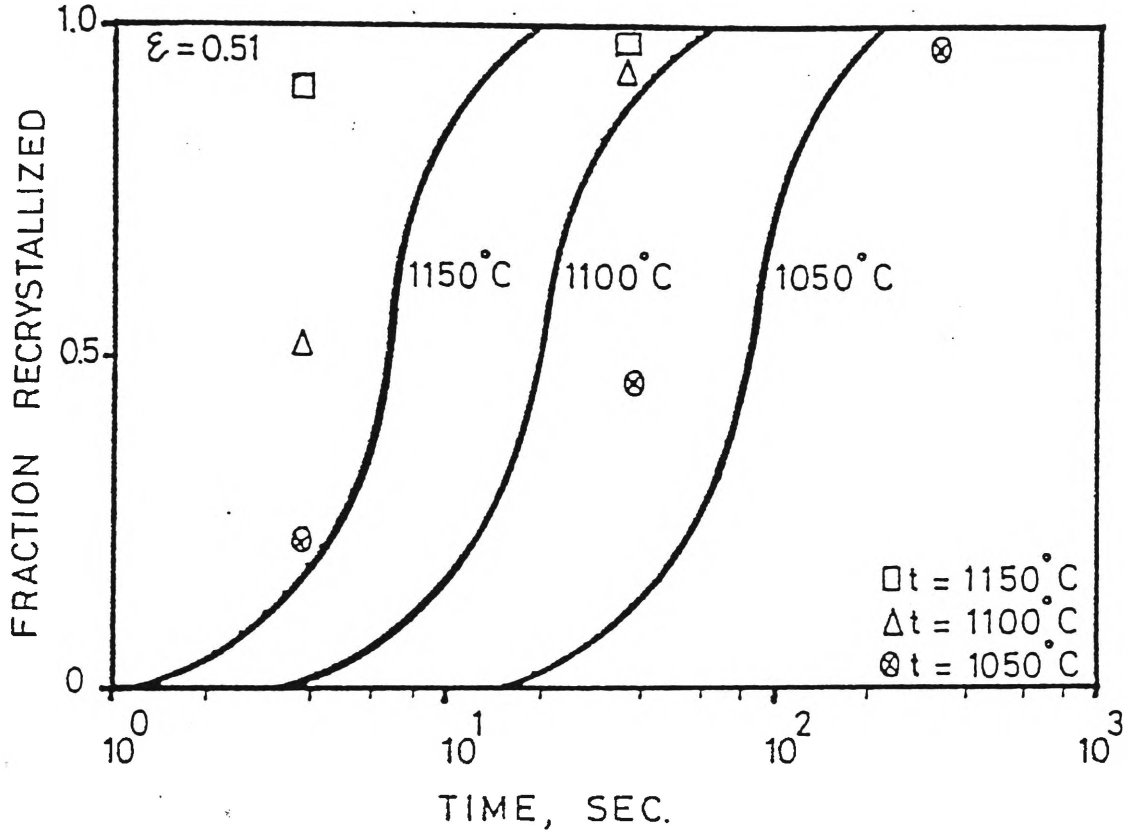

Figure 5.16. Kinetics of Static Recrystallization Following an AVRAMI Relationship [Eq.5.8] for Nb-V Steel at 1000 °C, 1050 °C and 1100 °C of Strain 0.51 Symbols Show Measured Values.

The symbols show measured values of much longer time from onset of recrystallization to complete recrystallization than Avrami's relationship for both, Nb-V and Nb-V-Ti steels. The time for complete recrystallization for Nb-V-Ti steel with true strain 0.51 (40% reduction) at a rolling temperature of 1050°C and above, may be regarded as similar to the predicted time. The observed time for complete recrystallization for Nb-V steel with true strain 0.51, is longer than the predicted time.

The onset of recrystallization for both steels, is earlier than Avrami's prediction, and for the same holding time after rolling, fraction recrystallized increased with increasing rolling temperature. The retardation of the progress of recrystallization after recrystallization start by microalloying addition can be attributed to their presence as solute and as precipitates or to a combination of the two. Also the increase in temperature can reduce precipitate contribution on retardation of the progress of recrystallization (52, 53).

5.6. GRAIN SIZE PREDICTION

The C-Mn Steel

When recrystallization is complete, the recrystallized grain sizes (d - rex) have been predicted for C-Mn steels by Sellars (4,45,50,51,52) using the following equation :

$$\text{d-rex} = D\, d_0^{0.67} \in^{-1} \qquad (5.9)$$

Where d_0 is initial grain size, $\in$ is true strain and D is constant (0.35, 0.5 and 0.83)

Equation (5.9) indicates that d-rex is independent of deformation temperature. Additionally, predictions of equation (5.9) depend strongly on the value of the constant D and to obtain the best predictions of the measured recrystallized grain sizes it may be necessary to change the constant D systematically with reduction (Table 5.3).

From Figure 4.12 the rolling coordinates 1050°C, 40% and 3 sec. lie near to this surface defining 100 % recrystallization and the measured grain size of 71 μm should correspond closely to the recrystallized grain size. Equation (5.9) predicts that at $\in$ = 0.51, d-rex = 43 μm, when using the constant D = 0.50, and d-rex is 72 μm when using constant D = 0.83 which is in good agreement with the measured grain size.

Since from equation (5.9) d-rex is independent of deformation temperature, the rolling coordinates temperature 1100°C and 40% reduction will also predict a grain size of 72 μm. The measured grain size of 82 μm is consistent with the completion of recrystallization with some grain growth within the quenching time of 3 sec. An alternative equation, by Roberts et.al (50,52,54), gives the following relationship of d-rex with temperature for C-Mn steel.

$$\text{d-rex} = 6.2 + 55.7\, d_0^{0.5}\, \epsilon^{-0.65}\, [\exp.(350.000/RT)]^{-0.1} \qquad (5.10)$$

For 40% reduction at 1050°C and 60% reduction at 1000°C, equation (5.10) predicts d-rex as 64 μm and 40 μm, respectively, are relativity close to the measured values of grain size of 71 μm and 49 μm in the C - Mn steel.

Table. 5.3. Predicted and Experimental Values of Recrystallized Grain Size in C-Mn Steel.

Reduction %	True Strain	d-rex (μm) Predicted by Equation 9			Predicted by Equation 10					Experimental (μm) *
		D=0.35	D=0.5	D=0.83	950°C	1000°C	1050°C	1100°C	1150°C	
60	0.92	17	24	40	35	40	45	50	58	40 - 80
40	0.51	32	43	72	48	55	64	69	83	47 - 115
20	0.22	73	94	166	75	92	104	114	138	68 - 165

* The range indicates d-rex values for the range of temperatures 950°C - 1150°C, used in the study.

The results of the investigation have been used to develop an empirical equation with temperature independency which is consistent with the measured recrystallized grain size. A plot of ln (d_{rex} / d_0) vs true strain (Figure 5.17) yields the following equation :

$$d\text{-}rex = d_0 \exp - (1.65 \in + 0.6) \qquad (5.11)$$

The Nb - V steel

Sellars also developed an equation for Nb steels (4,51,54).

$$d\text{-}rex = D\, d_0^{0.67} \in^{-0.67} \qquad (5.12)$$

where D is a constant (variously given as 0.66, 0.9, 1.1 and 1.86).

The predictions of equation (5.12) depend strongly on the D value and, similar to equation (5.9) for C-Mn steel, predicted values similar to the measured recrystallized grain size (d-rex) were obtained by varying the constant D with strain (Table 5.4). Using D = 1.1. Equation (5.12) predicts d-rex values of 36 μm and 54 μm, for reduction of 60% and 40% respectively, in good agreement with the measured grain sizes 46 μm for 60% reduction at 1050°C and 60 μm for 40% reduction at 1100°C (Figure 4.13)

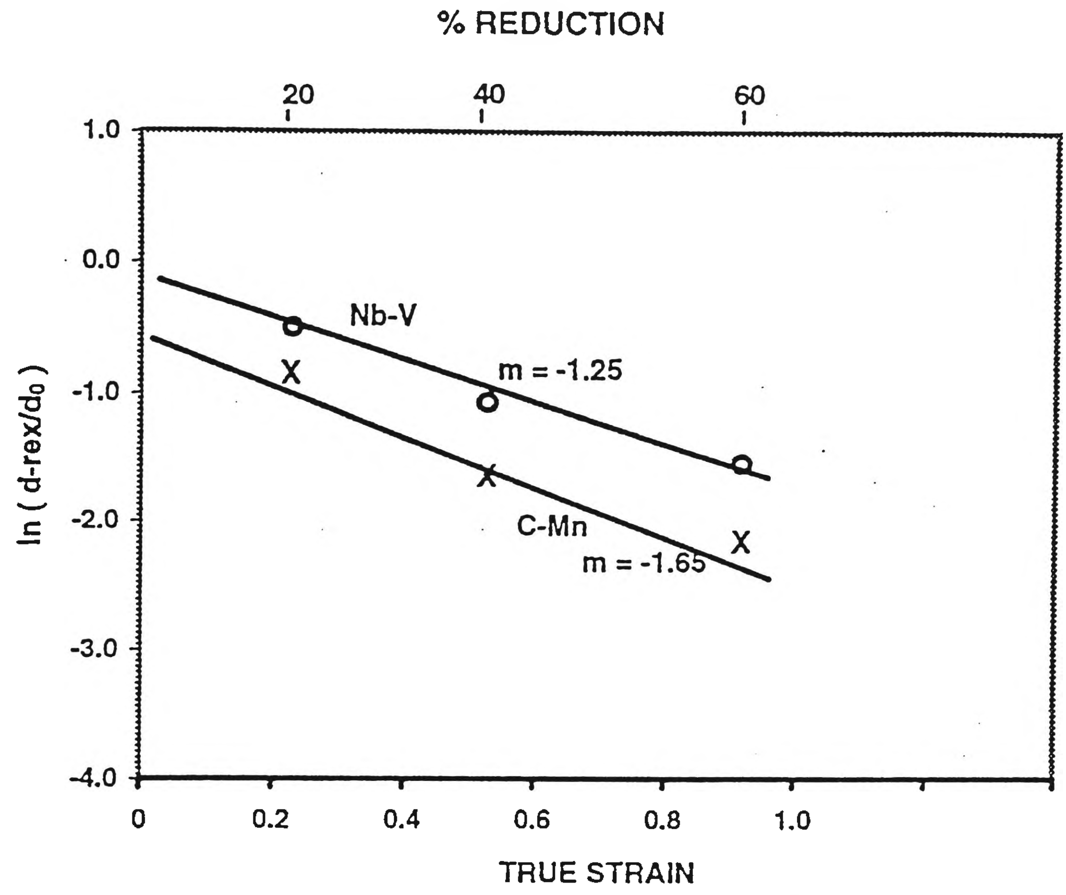

Figure 5.17. Recrystallized Austenite Size (drex) as a Function of True Strain (ε) in C-Mn & Nb-V Steels.

Table. 5.4. Predicted and Experimental Values of Recrystallizated Grain Size in Nb-V Steel.

Reduction %	True Strain	d-rex (μm) Predicted by Equation (12)				Experimental (μm) *
		D=0.66	D=0.9	D=1.1	D=1.86	
60	0.92	22	29	36	61	40 - 46
40	0.51	33	44	54	91	47 - 60
20	0.22	57	77	93	158	-

* The range indicates variations in d-rex values at various temperatures in the range 1050°C - 1150°C.

In a similar way to the C-Mn steel an empirical equation was developed from a plot of ln (d_{rex}/d_0) vs true strain (Figure 5.17) :

$$\text{d-rex} = d_0 \exp - (1.23 \in + 0.4) \qquad (5.13)$$

This equation also predicts recrystallized grain size values that agree closely with the experimental values, independent of temperature, as does equation (5.12) .

The Nb-V-Ti Steel

The best fit relationship of the dependence of initial grain size, time strain and temperature on the recrystallized austenite grain size for the Nb-V-Ti steel is given by the equation of Siwecki (52) :

$$\text{d - rex} = -1.25 + 24.4\,(V + Nb)^{-0.2}\, N^{-0.04}\, d_0^{\,0.25} \in^{-0.55} \exp\,(350.000/RT)^{-0.07} \qquad (5.14)$$

For 60% reduction at temperatures of 1100°C and 1150°C (Figure 5.17) the measured grain sizes are 34 μm and 35 μm, and are predicted by equation (5.14) to be 25 μm and 39 μm, respectively. Similarly, for 40% reduction at temperature of 1150°C, d - rex predicted from equation (5.14) is 42 μm, which is close to the measured value of 46 μm (Table 5.5).

From the above results it may be concluded that the equation by Siwecki is a good predictor of recrystallized grain size. The Nb-V-Ti steel slab used in the study had a Ti : N ratio less than the stoichiometric ratio, so all of the titanium would be expected to be available to combine with nitrogen to form titanium nitride particles, and of the remains of nitrogen would have an opportunity to combine with niobium and vanadium, and together, all these nitrides would retard recrystallization.

Table. 5.5. Predicted and Experimental Values of Recrystallized Grain Size in Nb-V-Ti Steel.

Reduction %	Strain ϵ	T °C	d - rex (μm)	
			Predicted Equation (14)	Experimental
60	0.92	1050	22	31
		1100	25	34
		1150	39	35
40	0.51	1050	33	40
		1100	38	43
		1150	42	46
20	0.22	1050	44	48
		1100	46	50
		1050	55	54

CHAPTER 6

CONCLUSIONS

6. CONCLUSION

The following conclusions were drawn concerning the steels investigated.

1. The grain coarsening temperature for the Nb-V steel was found to be about 1000°C, and for the Ti- Nb - V steel to be about 1050 ^{0}C .

   Austenite grain coarsening in the C-Mn steel occurred at a low temperature ($\leq$1000 ^{0}C) during reheating and this effect can be explained as due to the low solution temperature of AlN grain boundary pinning particles and therefore, low stability of AlN in austenite.

2. The grain coarsening temperature was increased by microalloying additions of niobium, vanadium and titanium.

3. The rate of recrystallization of the C-Mn, the Nb-V and the Nb-V-Ti steels increased with increase of rolling temperature or reduction. The recrystallized austenite grains in the C-Mn steel grew at a higher rate than in the Nb-V and the Nb-V-Ti steels for same rolling and holding conditions, which may be attributed to the relative absence of strong grain boundary pinning particles

4. The effect of microalloying additions was to increase the critical rolling reduction to induce recrystallization at a given temperature and holding time, but the critical rolling reduction decreased with an increase in rolling reduction and holding temperature. This effect is attributed to the strong pinning effect on austenite grain boundaries of alloy carbonitride particles.

5. The mean recrystallized austenite grain size increased with increasing temperature of deformation and this result agrees with theory. The Nb-V and the Nb-V-Ti steels exhibited only a slight increase in recrystallized grain size with increasing holding temperature , probably due to coarsening of alloying carbonitride particles.
6. The recrystallized grain size decreased continuously with increase in amount of reduction in all three steels investigated. The higher rolling reduction produced a high surface area per unit volume and a deformed structure which increased the reduction rate for recrystallization.

REFERENCES

REFERENCES

1. Ouchi, C. Sapei, T. Okita, T. Kozasu, I "Microstructural changes of Austenite During Hot Rolling and Their Effects on Transformation Kinetics", The Hot Deformation of Austenite, ed. J. Ballance, (1977).

2. Porter, D.A, "Phase Transformation in Metal and Alloys", Van Nostrand Reinhold Company Ltd, England, (1981).

3. Williams, J.G, "Production of Tough HSLA Heavy Plate Steels with a Moderate Controlled Rolling Process", Port Kembla, B.H.P, Port Kembla, Australia, (1989).

4. Pickering, F.B, "Physical Metallurgy and The Design of Steel", Applied Science Publishes, London, (1983).

5. Gladman,T., "Grain Refinement in Multiple Microalloyed Steels", Int. Proc. HSLA Steel, ed. G. Tither, Z. Shouhua, Beijing, China (1990).

6. Weiss,I., "The Influence of Niobium, Vanadium and Nitrogen on The Respone of Austenite to Reheating and Hot Deformation in Microalloyed Steels", in Thermomechanical Processing of Microalloying Austenite, Con. Proc, ed. A.J.De Ardo, AIME. Pittsburgh (1982).

7. Leslie, W.C, "The Physical Metallurgy of Steel", McGraw Hill, USA (1981).

8. Katsumata, M., Machida, M. and Kaji,H. , "Recrystallization of Austenite in High-Temperature Hot-Rolling of Niobium Bearing Steels", Thermomechanical Processing of Microalloyed Austenite, Pittsburgh. (1981).

9. Chandra, T., Misra, S. and Dunne, DP. "Static Recrystallization Kinetics of Hot Rolled Austenite in Titanium-Bearing HSLA Steels", ed. D.P. Dunne and T. Chandra, Proc. Int. Conf, Wollongong, Australia, (1984).

10. Sage, A.M., "An Overview of The Use of Microalloys in HSLA Steels with Perticular Reference to Vanadium and Titanium", Int. Proc. HSLA Steel, ed. G. Tither, Z. Shouhua, Beijing, China (1990).

11. Irvine, K.J. Pickering,F.B. and Gladman,T., I.S.I.J. " Grain Refined C-Mn Steels", (1967) 161.

12. Mangonon, P.L. and Heitmann,W.E., "Micro-Alloying'75", Proc. Int. Symp. HSLA Steel, Union Carbide Corporation, New York (1977),.

13. Kozasu,I., Ouchi,C., Sampei,I., and Okita, T., in "Micro- Alloying'75", Proc. Int, Symp. HSLA Steels, Union Carbide Coporation, New York (1977). P. 59-70

14. Ouchi, C., "Accelerated Cooling After Controlled Rolling HSLA Steel", Proc. Int. HSLA, ed. D.P. Dunne, T. Chandra, Wollongong, Australia (1984).

15. Meyer,L., Straburger,C., Schneider,C., "Effect And Present Application of the Microalloying Elements Nb, V, Ti, Zr and B in HSLA Steels", Con. Proc. Int. HSLA Steels, Beijing, China, ASM Int. (1985).

16. Ouchi,C., Okita,T., "Dynamic Recrystallization Behaviour of Austenite in Nb Bearing HSLA and Stainless Steel", ISIJ, 22 (1982).

17. Cuddy,IJ., "Grain Refinement of Nb Steels by Control of Recrystallization During Hot Rolling", Metal Transactions 15A, (1989), 87.

18. Weiss, I., and Jonas, J.J, "Interaction Between Recrystallization and Precipitation During the high Temperature Deformation of HSLA Steel", Metal Transaction, 10 A, (1979).

19. Weiss, I., and Jonas J.J, "Dynamic Precipitation and Coarsening of Niobium Carbonitrides During The Hot Compression of HSLA Steel", Metal Transaction, IIA (1980), 403.

20. Kozasu, I., Ouchi,C, Sampei,T and Okita, T., "Hot Rolling as S High Temperature Thermomechanical Process", in Microalloying 75, Union Carbide Corp, New York. N.Y. (1977).

21. White, J and Owen, T "Effect of Vanadium and Nitrogen on Recovery and Recrystallization During and after Hot Working", Metal Transaction, IIA (1980), 597.

22. Gladman, T and Pickering, F.B, "Grain-Coarsening of Austenite", Journa of the Iron and Steel Institute, June (1967).

23. Amin,R.K., and Pickering,F.B., Conf. Proc., "Thermomechanical Processing of Microalloyed Austenite", ed A.J. De. Ardo, GA. Ratz and P.J. Wray, AIME Pittsburgh, (1982), 1.

24. Tamura, I, Ouchi,C. Tanaka, T Sekine,H "Thermomechanical Processing of High Strength Low-Alloy Steels", Butterworth & Co. Ltd, London, (1988).

25. Cuddy, I.J. in "Thermomechanical Processing of Microalloyed Austenite", ed. A.J. De Ardo, G.A. Ratz and P.J. Wray. AIME Pittsburgh (1982).

26. Honeycombe, R.W.K " Transformation from Austenite in Alloy Steel", Metal Transaction, 7A, (1976).

27. Fuyu,S., and Wenxuan,C., “Disslocation And Precipitation of Nb, V, And Ti Carbonitride in HSLA Steels During Thermomechanical Processing”, Int. Proc. HSLA Steel, ed. G. Tither, Z. Shouhua, Beijing, China (1990).

28. Gauthier, G. and Lebon,A.B., "Micro-Alloying'75", Union Carbide Corporation, New York, (1977).

29. Tanaka,T., Tabata,N., Hatamura,T. and Shiga,C., "Three Stages of The Controlled-Rolling Process", in Mocro-Alloying 75, Union Carbide Corp, New York. N.Y. 1977.

30. Tanaka,T., "Four Stages of the Thermomechanical Processing in HSLA-Steel", Proc. Int. Conf, Wollongong, Australia ed. D.P. Dunne and T. Chandra (1984).

31. Katsumata,M., Machida,M. and Kaji, H "Thermomechanical Processing of Microalloyed Austentie", ed. A.J. De Ardo GA. De Ardo, G.A. Ratz and P.J. Wary, AIME. Pittsburgh, (1982).

32. Sellars, C.M., "Static Recrystallization And Precipitation During Hot Rolling of Micro-Alloyed Steels", Departement of Metallurgy, University of Sheffield, England (1981).

33. Sekine, H., Maruyama,T., Kageyana,H., in Conf. Proc, "Thermo-mechanical Processing of Microalloyed Austenite", ed.A.J. Deardo, Pittsburgh (1981).

34. Lebon, A.B., and de Saint-Martin,L.N. "Microalloying 75", Union Carbide Corporation. N.Y. (1977).

35. Jonas, J.J., "Mechanical Testing For The Study of Austenite Recrystallization And Carbon Nitride Precipitation", HSLA Proc. Int. Wollongong, Australia ed. D.P. Dunne and T. Chandra, (1984).

36. Jonas,J.J. and Akben, M., Metals Forum, 4 (1981).

37. Morrison, W.B., , J. Iron and Steel Inst., 210 (1972).

38. Malcom Gray, J., De Ardo,A.J., "Austenite Conditioning Alternatives for Microalloyed Steel Products", Con. Proc. Int. HSLA Steel in Beijing, China, ASM Int. (1985).

39. Tanaka,T., Enami, T., Kimura, M., Saito, Y. and Hatomura, T., " Formation Mechanism of Mixed Austenite Grain Structure Accompanying Controlled Rolling of Niobium Bearing Steels", Thermomechanical Processing of Microalloyed Austenite, Pittsburgh. (1981).

40. Subramanian, S.V., "The Effect of Nitrogen Content on The Evolution of Precipitates and on The Processing of Ti-Nb Beijing HSLA Steel Slabs", Con. Proc. Int. HSLA Steel in Beijing, China, ASM. Int. (1985).

41. Akben,M.G., Chandra,T., Plassiard,P. and Jonas,J.J., Acta Met, 32 (1984), 591.

42. Houghton,D.C., Weatherly,G.C. and Embury,J.D., "Thermomechanical Processing of Microalloying Austenite", Conf. Proc, ed. A.J. De Ardo, G.A. Ratz and P.J. Wray, AIME. Pittsburgh, (1982), 267.

43. Dunne, D.P., Chandra,T., and Misra,S., "Recrystallization of Austenite in A Hot Rolled Titanium HSLA Steel with Ti : N > 3.4", Con. Proc. Int. HSLA in Beijing, China, ASM. Int. (1985).

44. Ouchi,C., Sampei,T., Okita,T. and Kozasu,I., in "The Hot Deformation of Austenite", ed. J.B. Ballance, AIME, N.Y. (1977).

45. Tanaka,T, Enami,T., Kimura,M., Saito,Y. and Hatomura,T., "Thermomechanical Processing of Microalloyed Austenite", ed A.J. De Ardo, D.A. Ratz and P.J. Wray, AIME, Pittsburgh, (1982). 195.

46. Tanaka,T., Tabata,N., Hatomura,T. and Shiga,C., "Microalloying '75", Union Carbide Corporation, N.Y (1977).

47. Cuddy,C.J., Met. Trans, 12A (1981), 1313

48. Umemoto, M., Tamura,I., Hai Guo,Z., "The Effect of Austenite Grain Size on the Isothermally Formed Ferrite Grain Size", Con. Proc. Int. HSLA Steel in Beijing, China, ASM. Int. (1985).

49. Umemoto, M., Ohtsuka,H. and Tamura,T., "Prediction of Ferrite Grain Size Transformed From Thermomechanically Processed Austenite", HSLA. Proc. Int, ed. D.P. Dunne, T. Chandra, Wollongong Australia (1984).

50. Sellars,C.M. and Beynon,J.H., "High Strength Low Alloy Steels". ed. D.P. Dunne and T. Chandra, Proc. Int, Wollongong, Australia (1984).

51. Sellars,C.M., "Hot Working and Forming Processes", ed. C.M. Sellars and G.J. Davies, Metal Soc, Sheffield (1979).

52. Siwecki,T., "Modelling of Microstructure Evaluation During Recrystallization Controlled Rolling", ISIJ International vol : 32 No. 3 (1992).

53. Beynon,J.H., and Sellars,C.M., "Modelling Microstructure and Its Effects During Multipass Hot Rolling", ISIJ International vol : 32. No. 3 (1992).

54. Qilong,Y., Yongfu,L., Kunyu Z and Baurong,W. "A Special Static Recrystallization Phenomenon in V-Ti Microalloyed Steels", Int. Proc. HSLA Steel, ed. G. Tither, Z. Shouhua, Beijing, China (1990).